REPORT

EXPERIMENTAL EVALUATION OF THE ADHESIVE DEGRADATION POTENTIAL OF AQUEOUS CLEANING PROCESSES

To

Newark Air Force Base

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EXPERIMENTAL EVALUATION OF THE ADHESIVE DEGRADATION POTENTIAL OF AQUEOUS CLEANING PROCESSES

Final Report

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EXECUTIVE SUMMARY

The Aerospace and Metrology Center (AGMC), located at the Newark Air Force Base (NAFB) OH, repairs inertial navigation and guidance equipment for the United States (US) Air Force and other Department of Defense (DoD) components. Thousands of these delicate and sophisticated electromechanical devices are repaired each year at the Center. The current repair processes includes cleaning of these devices with chlorofluorocarbons and chlorinated hydrocarbon cleaners. Driven by safety and environmental concerns, AGMC is in the process of replacing these environmentally unacceptable cleaning agents with more environmentally friendly water-based detergent. A recent Presidential Executive Order has been issued reinforcing the need to implement replacement cleaning systems with a revised implementation date of 1995.

An experimental program has been completed which exposed selected epoxy and sealant materials to commercially available detergents in a statistically designed exposure matrix. Ultrasonic and immersion cleaning over a 12-cycle simulated life cycle at room temperature, 135 F, and 190 F was studied. Bulk effect and interfacial bonding responses were determined on the exposed samples. Freon 113, 1,1,1-trichloroethane, and pH-controlled water were used as controls.

Significant degradation was not observed for epoxy-detergent combinations. For epoxies, aqueous cleaning introduced comparable and generally smaller changes in adhesive properties as compared to using Freon 113 and 1,1,1-trichloroethane. A solvent substitute for these two halogenated solvents, PF Degreaser, performed similarly, to the halogenated solvents inducing similar degradation. Careful selection of the adhesive-detergent combination can reduce degradation for epoxy adhesives below the levels caused by the halogenated solvents. Based on results of this study, aqueous cleaning with selected detergents at temperatures less than 135 F is recommended as a replacement for halogenated solvents.

Process conditions, with the exception of high temperature, were found to be insignificant variables for causing degradation. Life cycle cleaning was also shown to be either a main effect or an interactive effect with temperature. Versaclean and LCA4/BA5

Adhesive were studied further using a full factorial design. Analysis indicated that this combination is particularly stable, showing no significant degradation over the entire process spectrum. Detergents Versaclean and EZE 240 were shown to be the best selection for general cleaning.

Sealant materials did not tolerate cleaning well, exhibiting poor dimensional stability and large increases in weight (10-20%) due to absorption of the cleaning solution. Also discovered was an increase in hardness which could restrict the sealants function. Sealant materials do not tolerate aqueous cleaning well and significant degradation of these types of materials was observed.

Chemical analysis performed on epoxy adhesives indicates that degraded samples have absorbed the detergents they were exposed to. Minor hydrolysis, changes in functional groups, and formation of carboxylic acid groups were observed. Thermal analysis was unable to determine specific trends related to degradation but did show that thermally degraded samples do not act significantly differently than unexposed materials.

No overall interactions were observed relating interfacial and bulk degradation. Epoxy degradation when observed for a specific adhesive characteristic was generally unique for a particular adhesive-detergent combination. Therefore, application-critical performance factors must be used to guide selection of cleaning processes rather than applying a blanket set of rules.

It has been shown in this study that aqueous cleaning can be successfully substituted for Freon 113 and 1,1,1-trichloroethane solvent cleaning with a minimum impact on AGMC operations. Restricting high temperature and lengthy exposures are the only process restrictions that must be observed. Recommended adhesive-detergent combinations have been developed, and appropriate cautions for use and potential problems have been identified. These recommendations, coupled with AGMC's application expertise, should eliminate potential degradation. The detergent systems appear to be temperature stable and should be useful over long periods of time, further reducing overall chemical usage at AGMC.

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EXPERIMENTAL EVALUATION OF THE ADHESIVE DEGRADATION POTENTIAL OF AQUEOUS CLEANING PROCESSES

Final Report

1.0 INTRODUCTION

The objective of this test program was to experimentally evaluate the degradation to adhesives caused by selected aqueous cleaning operations associated with precision cleaning of inertial guidance components in a repair and maintenance environment. This test program covered the evaluation of various cleaning methods and their effect on representative adhesives to identify specific material-process combinations in which significant adhesive degradation would not occur and others in which it would occur. The goal of the program was to provide AGMC with information required to select appropriate processes and aqueous cleaning agents to use as alternatives for existing CFC and halogenated hydrocarbon cleaning processes. The scope of the project was to design and conduct a series of experiments, evaluate the results, and report the potential for degradation that may result from replacing the solvent systems currently used for precision cleaning with various water-based detergent cleaners and cleaning processes.

An overview of the program's work elements is shown in Figure 1. This program was an initial step in developing a screening tool to determine which combinations of detergents and adhesives can be used without degradation of adhesive bonds in an aqueous cleaning operation of inertial components serviced by AGMC.

The adhesives and detergents were sorted and classified into chemical and product families. Identification of the mechanism of degradation and specific chemical components of the cleaning agents which cause degradation were studied by photo-acoustic FT-IR and thermal analysis.

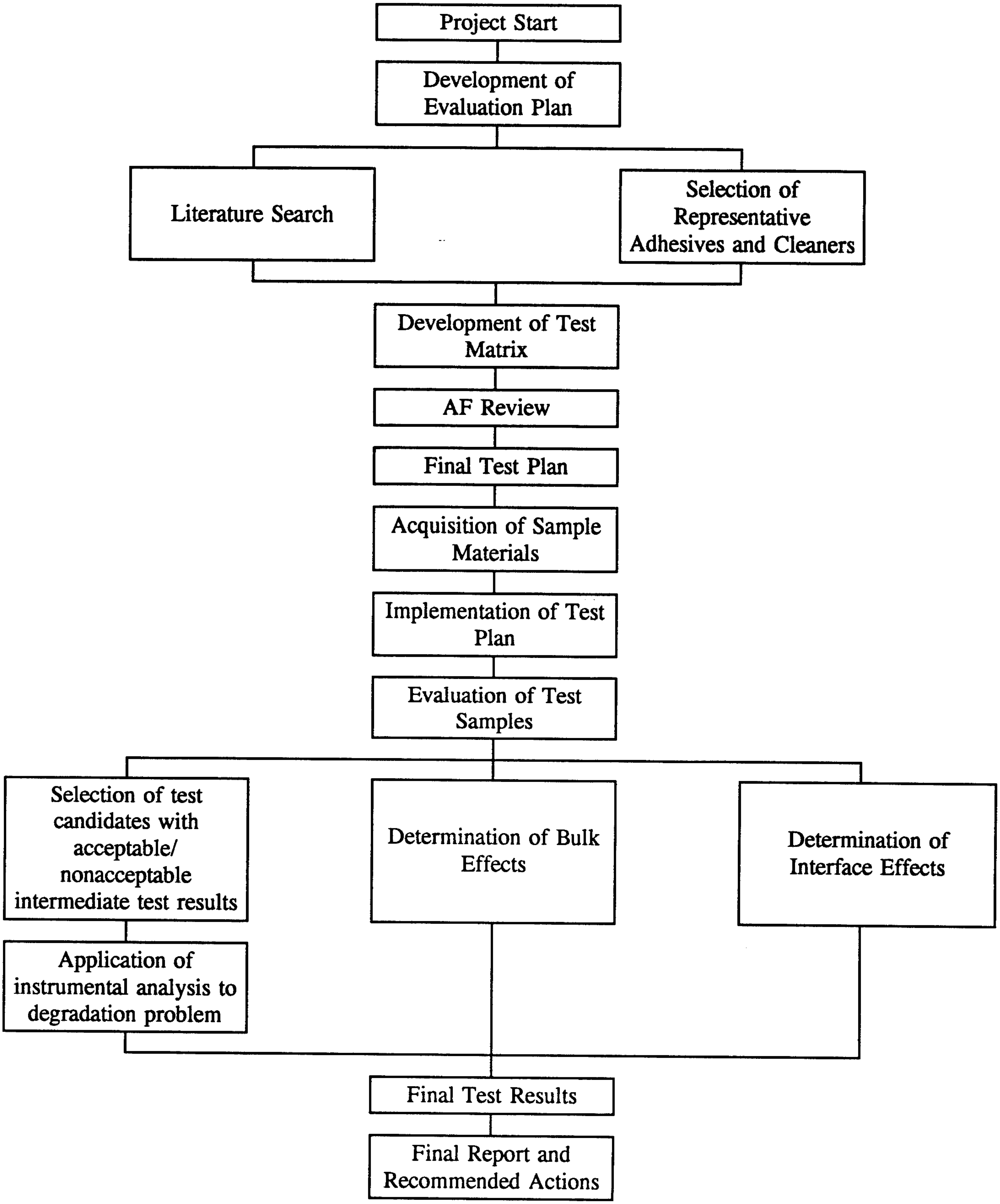


Figure 1. Overview of Program Activities

1.1 Background

AGMC has developed some experience with the use of detergent cleaners and has observed that some degradation is occurring in epoxy bonded structures along the bondline. The nature and extent of the observed degradation presently occurring and its future degradation potential are unknowns and of great concern. (The commercial market does not cycle repaired systems through their cleaning cycle and is thus only concerned with single exposure effects.) Additionally, commercial electro-mechanical systems typically are not designed for longer service life cycles than 10 years. AGMC routinely does repetitive repair cycles on components and service lives are frequently much greater than 10 years. Therefore, repeated cleaning cycles would occur, and it is necessary to determine the type and extent of adhesive degradation associated with repeated use of and exposure to aqueous cleaners and associated processes. Degradation of the adhesive, if allowed to occur, could adversely affect the reliability, life cycle, and maintainability for these repaired devices.

The program test plan was designed to experimentally evaluate the degradation potential of aqueous cleaning methods on cured polymeric structural adhesives. The primary type of adhesives studied is epoxies, which are two-component, low-temperature, heat-curing systems. Sealants which are also used as adhesives by AGMC have also been included. All the adhesives in this study are designed for use in inertial components as either OEM or repair materials.

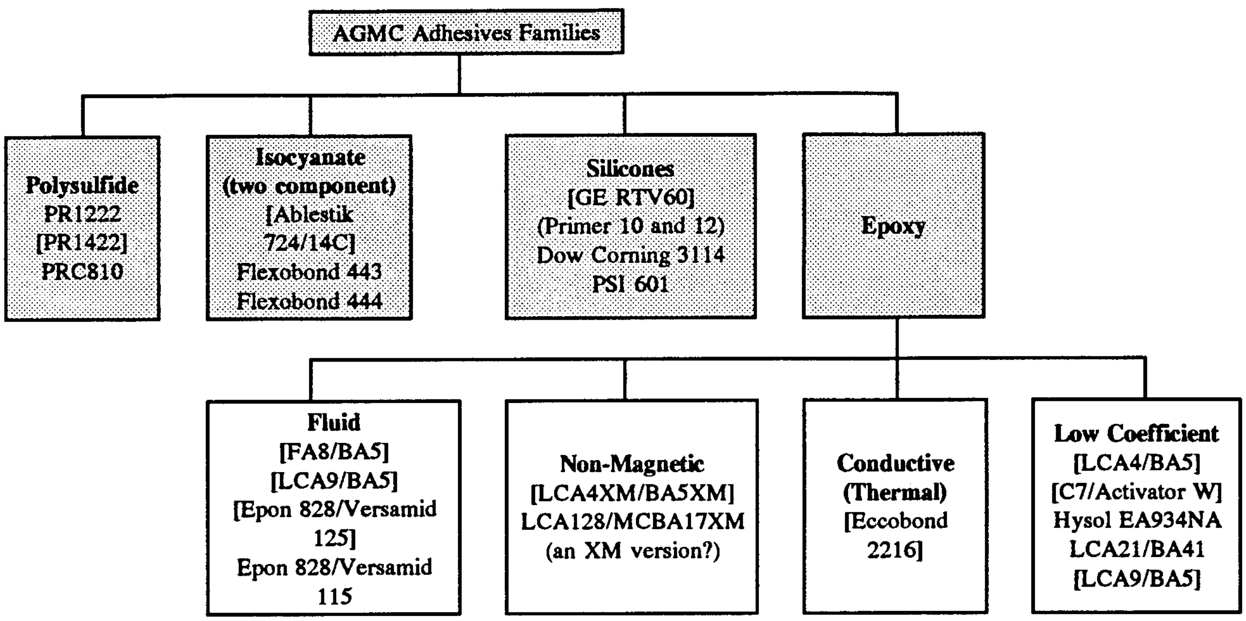
To accomplish the program's objectives, a statistically designed experiment was conducted in which cured lap shear and cast bulk samples were exposed to various cleaning agents and methods and then evaluated for performance changes resulting from the cleaning operation. The cured samples were evaluated after a single exposure and lifetime exposure consisting of twelve cleaning cycles. During this experimental exposure matrix, selected samples representing stable and degraded performance were further evaluated using instrumental chemical analysis to determine if the cause and extent of the degradation could be identified and quantified.

1.2 Approach

The objective of this study was to assess the potential adhesive degradation problems which may be associated with the use of water-based detergent cleaners used to clean electromechanical parts. This project used the AFMC Design Engineering Program (DEP) to implement the goals of the Air Force Reliability and Maintainability (R&M) 2000 Program of making R&M a primary consideration in systems or process modifications by assessing, in a rapid and cost effective manner, the effect of the proposed process modification on R&M prior to its implementation. The study identified the basic material/process combinations which will or will not induce adhesive degradation, alerting AGMC to avert degradation-causing conditions. Because of the large number of process and material combinations involved in this study, a statistically designed exposure matrix was developed. This exposure matrix developed the maximum amount of data for the minimum amount of effort. Without the use of a matrix, the program would have been physically impossible to conduct.

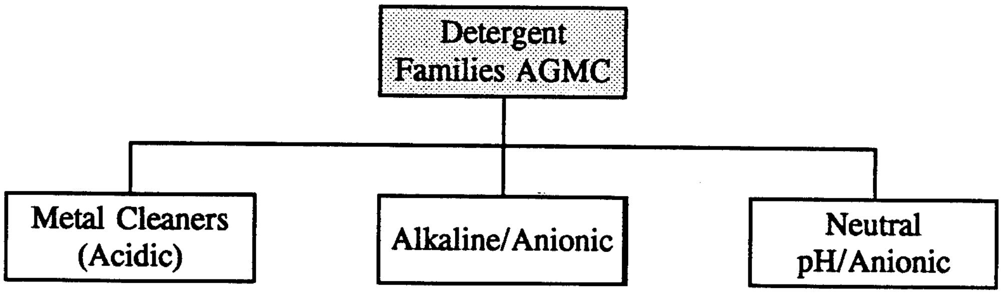
2.0 EXPERIMENTAL SELECTION OF CANDIDATES

Battelle reviewed the adhesives and cleaning agents being used by AGMC and selected representatives for each chemical type. AGMC supplied selected products in sufficient quantities to support program activities while the remainder were purchased. A technical representative visited AGMC and discussed AGMC practices and protocol for using adhesives and cleaning agents. This information was used to develop and guide Battelle's selection of test candidates for the exposure matrix. See Figures 2 and 3.



XM = nonmagnetic XM
[ ] = selected for study
( ) = recommended primer system

Figure 2. AGMC Adhesive Families



Types of surfactants:

Anionic—most common type in cleaners, ionizes to yield negatively charged species, i.e., $-OSO_4$, SO_4, carboxylic groups, PO_4

Cationic—rarely used in a cleaner ionizes in solution to yield positively charged species, i.e., quaternary amines

Nonionic—commonly used in cleaners as a co-surfactant, high detergency and low foaming, does not ionize in solution; long chains of ethylene and propylene oxides are functional portion of system

Figure 3. AGMC Detergent Families

Selection of the adhesives and detergents used in this study was guided by input from AGMC and interpretation of supplier literature. Test candidates were selected using a priority system shown below. The highest priority, current use in production, guided the majority of the selections.

Priority	Criteria
1	In production use at AGMC
2	Recommended for electronic cleaning and no toxicity problems
3	Recommended for electronic cleaning, has limited toxicity restrictions
4	Model surfactants used as a simple or non-formulation product

A list of the detergent candidates selected can be found in Table 1. These eight detergents were diluted with distilled water to the concentrations indicated in the table.

Table 1. Detergent Test Candidates

Detergent	Variable No.	Lot No.	Class	Recommended Level Light Soil, General Use	Volume Use Level (mid-level) Maximum 5%	Supplier
Versaclean	1	TSRN 80100-145P	Detergent	1:10 to 1:60 1:30 gen.	3.3%	Fisher Scientific
Brulin 8156D	2	GD027	Detergent	5%	5%	Brulin & Co.
EZE 240	3	G230022	Detergent	None	2%	EZE Products
Intex 8125	4	F229139	Detergent	None	2%	EZE Products
MSI 1025	5	I916774	Detergent	None, application dependent	2%	Magnosonic Systems
Oakite Liq. Det. #2	6	HA213	Detergent	2.5%	2%	Oakite Products
PF Degreaser	7	PF2703	Solvent replacement	Neat	100%	P-T Technologies
Citranox	8	09726PV	Metal cleaner	1-2%	2%	Alconox

This concentration is referred to hereafter as the standard concentration. A complete listing of the detergents considered for the study can be found in Appendix H.

All the adhesives selected (Table 2) were two components with the exception of Ablestik 724/14C, which was supplied in a premixed, frozen form. Therefore, the adhesives were mixed as needed throughout the operation of the matrix. The variability this introduced into the matrix is discussed in Section 4.0 and is contained as part of the Group effect. This effect did not affect the matrix significantly. It does suggest that if great care is not taken in mixing the two components properly, changes in resistance to cleaning exposures is expected.

The detergents used in this study are composed of three types: acid containing metal cleaners (Citranox), PF degreaser (a halogenated solvent alternative), and anionic formulated systems which comprise the remaining products. Anionic detergents are composed of many different specific chemicals which ionize above pH 7.0 to form ionically charged groups. An analysis of the available MSDS sheets for the detergents used in this study indicates that Intex 8125 contains a high boiling glycol ether which is not present in the other materials. The lack of compositional data from the MSDS sheets for both detergents and adhesives prevented further analysis.

Table 2. Adhesive Test Candidates

Adhesive	Variable No.	Lot No.	Type	Mfg. Recommended Mix Ratio		Recommended Cure Schedule	Supplier
				A	A		
FA8/BA5	1	102/211	Fluid-epoxy	100	13.5	2 hrs at 200 F	Bacon Industries, Inc.
LCA9/BA5	2	157/211	Fluid/epoxy	100	4.5	"	"
LCA4/BA5	3	300/211	Low coefficient/epoxy	100	4.5	"	"
LCA4XM/ BA5XM	4	168/92	Non-magnetic low coefficient epoxy	100	4.5	1 hr at 120 F + 2 hrs at 200 F	"
Ablestik 724/14C	5		Isocyanate/ Urethane			165 F/ 5 hrs	Ablestik/National Starch Inc.
PR1422B	6	Pt B = EF0B01301 Pt A = same	Polysulfide 93% NV	7.5	1.0	room temp. 32 hrs, 48 full	Semes Bancroft
Adhesive C-7/ Activator W	7	Pt A = HA0376 Pt B = IA0407	Low coefficient epoxy	1.0	1.0	48 hrs room temperature	Morton International Inc.
GE RTV60	8		Silicone sealant RTV			24 hrs room temperature	General Electric Corp.
Epon 828/ Versamid 125	9	05PHJ72/2E8 240	Fluid epoxy	1.0	1.0	24 hrs room temperature	Miller-Stephenson Chemical Co.
Eccobond 2216	10		Thermally conductive				3M, Inc.

3.0 EXPERIMENTAL AND MATRIX DESIGN AND IMPLEMENTATION

This section describes the statistical design selected and the analysis that was performed for the experimental evaluation of the potential adhesive degradation due to aqueous cleaning processes. This experimental program has been designed to extract as much information as possible from the experimental trials performed.

3.1 General Matrix Background

A statistical design for an experiment provides a "blueprint" for the trials to be run and the data to be collected. It specifies the values of the independent variables corresponding to which observations of the dependent variables (also called responses) were observed. In this evaluation, independent variables, which were controlled experimentally, included adhesive, detergent, cleaning method, and temperature. The independent variables are described in Table 3. X_1 represents the type of adhesive and has ten levels. That is, for each trial, X_1 is one of ten adhesives. X_2 is detergent and has twelve levels, including the following controls (See Table 7): distilled water (C1), 1,1,1-trichloroethane (C2), Freon-113 (C3), pH-adjusted distilled water (C4). X_3 is cleaning method and has three levels: sonication at 20 kHz for 5 min using equipment equivalent to that used by AGMC, sonication at 67 kHz for 5 min., and soaking for 60 min. The fourth independent variable, X_4, is the temperature of the bath at the start of the cleaning process. Temperatures of 72 F, 135 F, and 190 F were used. Because of the number of trials required for this evaluation, the experiment was run in two blocks. X_5 is included in the design as the blocking variable to account for this. See Table 4 for the matrix implementation schedule. In the original design, each block consisted of 60 trials run in 3 groups over a period of 6 weeks. Upon completion of Block I the design of Block II was modified and extra trials were incorporated.

The experimental design selected is a fractional factorial design consisting of 120 trials for each initial exposure and life-cycle testing. Fractional factorial designs are powerful in screening for important effects and are typically used when the number of

Table 3. Independent Variables

Variables	Description	Number of Levels
X_1	Adhesive — Each level represents a distinct adhesive to be tested.	10
X_2	Detergent and Control Solutions — Each level represents a distinct cleaning solution, including 8 selected detergents plus controls.	12
X_3	Cleaning Methods — The two methods of cleaning are sonication and soaking. Nested within X_3 is sonication which is frequency dependent. (UL) = sonication at 25 kHz (UH) = sonication at 67 kHz (S) = soaking	3
X_4	Temperature — Levels selected are 72 F, 135 F, and 190 F to cover a feasible range for this quantitative variable.	3
X_5	Block — Each of the 2 blocks contains 60 trials.	2

Table 4. Matrix Implementation Schedule

		Trial Numbers
Block I Cycle Time 6 weeks	Group A	1-20
	Group B	21-40
	Group C	41-60
Block II Cycle Time - 6 weeks	Group D	61-81
	Group E	82-102
	Group F	103-124

Reference Appendix B

possible combinations of factor levels is prohibitive. They permit the efficient elucidation of important effects. (See Box, Hunter & Hunter in Section 7.0, *References*.)

A summary of the dependent variables measured in this study can be found in Table 5.

Table 5. Exposure Matrix Dependent Variables (Measured Responses)

1.	Weight loss or gain	Measured at selected intervals over exposure life cycle
2.	Dimensional Changes	Length, width, thickness
3.	Shore Hardness	ASTM D2240
4.	Visual Changes	
5.	Lap Shear	ASTM D1002
6.	DSC/T_g	
7.	TMA	Selected samples[A]
8.	FT-IR	Selected samples[A]

[A] samples showing significant degradation (3x of control) were selected

3.2 Matrix Design Elements

The specific design that was used is presented in Appendix A. The design is, as much as possible, orthogonal based on main effects and does not anticipate any statistical interactions between detergent and adhesive. The 10 adhesives are designated by single digits to be assigned to specific adhesives based on selection by Battelle and the Air Force. Similarly, the eight detergents are designated 1 through 8, while the five control solutions are shown as C1, C2, C3, C4A, and C4B. (C4A and C4B taken together were given equal representation with the other controls in the design.) In addition to the exposure conditions in the design, unexposed control specimens were prepared and evaluated. These are discussed in greater detail in Section 4.1. The levels of X_3 are designated UL and UH for the lower and higher frequency sonication, respectively, and S for soaking. $X_4 = 1$

corresponds to an initial bath temperature of approximately 72 F (room temperature), while $X_4 = 2$ implies a temperature of 135 F, and $X_4 = 3$ is 190 F. The trial number is indicated in the left column. One trial involves setting the independent variables at the levels indicated in a single row of the matrix, executing the experimental procedure, and recording data as specified in the procedure. The general testing schedule is shown in Table 6. Unexposed control specimens of each adhesive type were evaluated at time intervals corresponding to initial and life-cycle evaluations.

Table 6. Testing Schedule

Time	Bulk Testing			Interfacial	Thermal Analysis
	% Water	Bulk* Hardness	Physical* Dimensions		
Exposed Samples					
1st Exposure Post-Air Dry (5.2.4e)	X				
1st Exposure Post-Drying Cycle (5.2.5)	X	X	X	X	X
Life Cycle Post-Air Dry (5.2.4e)	X				
Life Cycle Post-Drying Cycle (5.2.5)	X	X	X	X	X
Unexposed Samples					
1st Exposure Drying Cycle (5.2.5)	X	X	X	X	X
Life Cycle Drying Cycle (5.2.5)	X	X	X	X	X

* pre-exposure baseline measurements will be made

The design was run in 2 blocks, each comprising 6 weeks of experimentation. (See Section 4.1.2 for further details.) Results from the first block were analyzed in a preliminary manner after completion of the first 60 trials (Block I). Insights gained during the first block suggested several changes in the design. The between-sample variability was sufficiently low to discontinue the practice of running triplicate samples for lap shear in favor of duplicate lap shears. (Duplicate bulk samples were run throughout the program.) The

design was reevaluated and modified before proceeding to Block II to include a full factorial exposure of LCA4/BA5 to VersaClean detergent.

The design was chosen to study selected factors and their effect on bond performance. Other factors that may affect bond performance include cavitation in the bond, degree of cure or age of the bond, cleaning time, sonication power imparted to the solution, mass of material in the batch, and concentration of detergent solution. These are not varied within the design; they were held constant as much as possible throughout the experiment. The number and type of samples in each batch were recorded as part of the data collection. The analysis of the data is discussed in detail in Section 4.

Several controls have been included in the matrix to serve as benchmarks. A set of detergent controls is listed in Table 7. Freon 113 and 1,1,1 trichloroethane (hereafter referred to as 1,1,1) were included as representing the current cleaning solvents in use at AGMC. Distilled water by itself and acidified and alkaline pH adjusted distilled water are the three water based controls. The amount of acid and base used to make the controls was based on titration of representative acid and alkaline detergents selected for the study. The pH and surface tension of the detergents as used in the study can be found in Appendix H.

Table 7. Detergent Controls

Controls	Description
C_1	Distilled Water
C_2	1,1,1 Trichloroethane
C_3	Freon 113
C_4A	Acidified Distilled Water, HCl
C_4B	Alkaline-Adjusted Distilled Water, NaOH

A standard solution of Detergent 8, Alconox's Citranox, was titrated with NaOH and an acid number of 3.8 was determined using ASTM D3643. Based on this data, a hydrochloric acid/distilled water simulant of Citranox was prepared by adding 6.37 gms of concentrated HCl to 1,000 gms of distilled water. The resulting solution's pH was 2.0

versus 3.2 for Citranox. This discrepancy in pH indicates that the acidic functions in Citranox are not fully ionized when diluted with water, yielding a higher pH solution than expected. The acid number is based on titration with a strong base which would be expected to ionize all of the acid groups present.

Brulin 815GD was selected as a representative alkaline fortified detergent. Using standard titration techniques, a standard solution of 815GD was titrated with HCl. Based on this titration, it was calculated that 4 gms of NaOH per 1,000 gms of H2O would yield a solution with the same level of titratable base present. As the case with the acidified control, the alkaline adjusted control had a higher alkaline pH value than 815GD. This again indicates that the actual solutions of the detergents are not totally ionized and that the strong ionizing titrant used in the analytical determination yields a higher value.

The independent variables used in the matrix are listed in Table 3. Independent variables are variables which can be set at pre-determined levels which are not linked or influenced by other independent variables. Two classes of independent variables are present: process and material. The process variables are the conditions the samples were exposed to physically and include temperature, type of exposure, and the number of cycles the sample had been exposed to. The material variables are type of adhesive and detergent. The detergent variable does not include the control cleaning systems and contains eight detergents.

The dependent variables for this matrix are listed in Table 5. Dependent variables represent measured response of the effects that are caused by the actions of the independent variables. Three classes of responses are represented:

- bulk effects e.g. weight changes
- interfacial effects or lap shear values
- chemical analysis
  - — glass transition temperature(DSC)
  - — thermal dimensional changes(TMA)
  - — functional group analysis(FT-IR)

The exposure processes used in this program were modeled after the current practices used at AGMC. A summary of the cleaning processes used in the matrix can be found in Table 8. In all cases the exposure cycle was completed with a rinsing and drying of the sample. (Appendix B provides further details.) The immersion cycle was performed in a batch process mode and covered the exposed sample completely with liquid at the selected temperature for a one hour cycle time.

Table 8. Exposure Processes

Exposure Type	Duration	Type of Equipment	Comments
Immersion	1 hour	Convection air oven, stabilized for 16 hours at desired temperature	Batch processing
High frequency ultrasonic cleaning	5 min	Mettler Model ME4-6, samples suspended in tank	85 watts/1 gal; 67 KHz nominal, frequency 40-70 KHz
Low frequency ultrasonic cleaning	5 min	Sonicator®/Heat Systems - Ultrasonic, Inc., Model 385, samples suspended around central transducer in a jacketed tank temperature regulated	Standard tip; 100 watts output, 200 watts/gallon used for exposure; 25 KHz nominal

The ultrasonic exposure method also completely covered the sample with liquid at the proper temperature followed by five minutes of exposure to the selected frequency. Two frequencies were evaluated, 25 Khz and 67 Khz. The low frequency exposure was accomplished using a Heat Systems, Incorporated Model 385 Sonicator while the higher frequency exposures were conducted using a Mettler ME4-6. The original AGMC supplied 40 Khz Delta Sonic bath failed immediately when turned on. This required the substitution of the Mettler ME4-6 which operates in a non-scanning mode from 40 to 70 Khz with nominal output at 67 Khz. This operational output is provided by supplying the transducer with a "dirty" electronic signal allowing the transducer to produce a wide frequency range. The frequency does affect the size of the ultrasonic bubbles produced. Information from Mettler indicate that 10-20 micron bubbles are produced at 67KHz and 50 micron bubbles at 25 kHz. The cleaning power is related to the bubble size as smaller bubbles are able to enter and clean smaller surface defects. Power density for this program

was calculated based on the total power consumption of the unit per volume unit area. This practice is common in the industry which has not developed a reliable power density measurement technique. A crude method used in production quality control by Mettler places a small vial containing light metal balls into the filled bath and then measures the height the balls are raised by the ultrasonic fields actions. This measurement is related to other units as a dimensionless indication that the unit is performing similarly to other units.

3.3 Matrix Operational Overview

The overall schedule for this testing program was based on an experimental matrix of 120 trials consisting of two equal blocks of sixty trials each. Each block was run in groups of 20-24 trials each for three consecutive weeks. The exposure cycle followed in this program is shown in Figure 4. Lifetime exposure tests were conducted concurrently with ongoing activities. Each block required six weeks to complete. After completing Block I, Block II was conducted by repeating the process. Only minor modifications between Block I and Block II activities were tolerated to minimize experimental error.

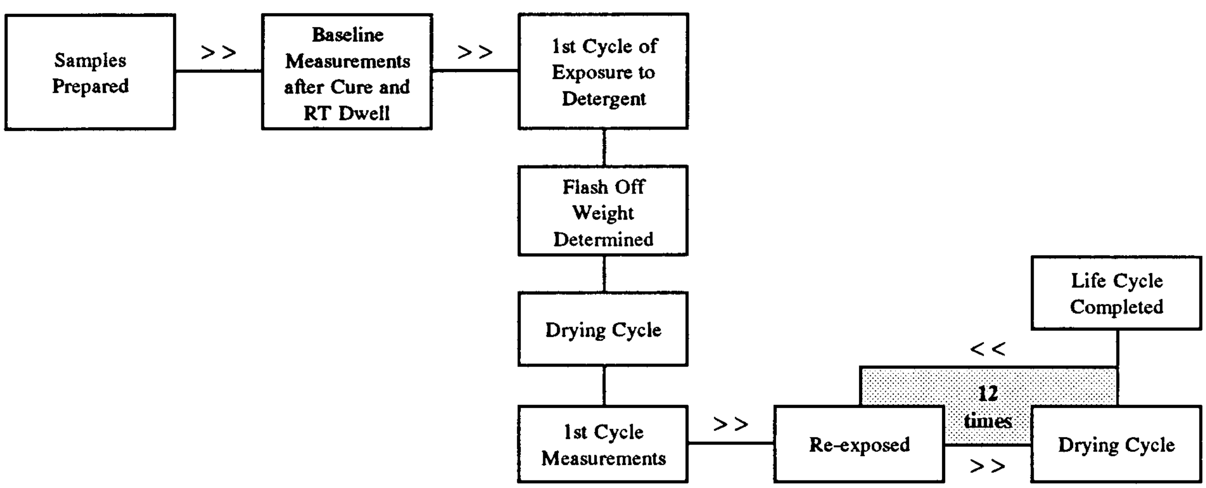


Figure 4. Exposure Cycle

The procedures performed under this effort and described in Appendix B follow ASTM and MilSpecs whenever possible. The procedures were not intended to produce qualification or acceptance type data but were designed to produce the data necessary to execute the test matrix and evaluate the overall degradation potential of aqueous cleaning on adhesives. Refer to Section 7, *References*, for ASTM methods cited in the test plan.

The statistical design of the experiment from the approved test plan was discussed in the previous section. (Also see Appendix A.) Often, real-world constraints within the laboratory prohibit running an idealized design. In the case of this study, certain constraints were imposed on the design during the planning stage in recognition of this reality. For example, it was the goal to randomize as much as possible with the understanding that complete randomization of conditions was infeasible. Thus, the design restricted the number of adhesives per group of trials to three. Moreover, there was a limitation on the number of detergent-method-temperature combinations that could be scheduled within a group. These two factors imposed constraints on the original design. In addition, unforeseen[1] events, such as the fact that the flash point of Detergent 7 (PF Degreaser) is below the temperature level 3, resulted in the need for modification of the design after the experiment began.

During the course of the experiment, lessons learned were incorporated in plans for additional trials. Some specimens did not hold up under the experimental conditions. Certain lap-shear samples separated in the bath, and some bulk samples distorted too much for meaningful physical measurement. For these reasons, responses were not obtained for every trial.

Appendix D presents the matrix of experimental trials as run. Significant changes were made in Block II when compared to the original design. The matrix indicates numerical values for all variables. An explanation of how these map to the design is in order. The first column contains the trial number, which generally corresponds to that in the

[1] Unforeseen because the selection of detergents was made after the original design was finalized.

design, with the following exception. Each time an adhesive was prepared for lap-shear samples or bulk specimens, control samples were also prepared. These samples, stored at 72 F and 50% relative humidity, received no exposure but were tested at the same time as their corresponding experimental samples. Four-digit trial numbers correspond to these controls. The first digit of the trial number for control samples indicates the group number. The next two digits indicate adhesive number, and the final digit is a zero. This numbering scheme permits easy identification of control samples.

Trials were run in two blocks, each block containing three groups. Block I included Groups A, B, and C, while Block II included Groups included D, E, and F. In the design matrix, these groups were denoted as Week 1 through Week 6 and correspond to Groups 1 through 6 in Appendix A.

Another convention adopted for presentation and management of the data was to reserve the number 0 for an unexposed control. Thus, Adhesive 0 in the original design was renumbered as Adhesive 10 (Eccobond 2216). Detergent 0 signifies no detergent exposure. The detergent controls were then assigned sequential numbers 9 through 13. Method 0 became no exposure, as did Temperature 0. Table 9 provides the key to the numbering scheme.

In some of the analyses, temperature was treated as the continuous variable that it is. In this case, temperature was normalized between -1 and +1, and temperature level 0 was treated like temperature level 1 because both are approximately room temperature.

All trials were replicated at least once. Groups 1, 2, and 3 of the lap-shear specimens were run in triplicate, while Groups 4, 5, and 6 were run in duplicate. Bulk samples were run in duplicate.

Table 9. Legend for Variable Levels

Variable	Assigned Number	Key in Design	Description of Level
Detergent	0	---	No detergent exposure
	9	C1	Deionized water
	10	C2	1,1,1-Trichloroethane
	11	C3	Freon-113
	12	C4A	Acidified distilled water
	13	C4B	Alkaline-adjusted distilled water
Method	0	---	No exposure
	1	UL	Sonication at 25 kHz
	2	UH	Sonication at 67 kHz
	3	S	Soaking
Temperature	0	---	No exposure
	1	1	72 F
	2	2	135 F
	3	3	190 F
Group	1	A	Block I, Week 1
	2	B	Block I, Week 2
	3	C	Block I, Week 3
	4	D	Block II, Week 4
	5	E	Block II, Week 5
	6	F	Block II, Week 6

4.0 RESULTS

4.1 Introduction

This section presents the results of the analysis of the data collected under this adhesive evaluation project. This introductory subsection describes the responses evaluated and general information about the analysis process. These lap shear results are presented in Section 4.2, while those from the bulk data related to physical changes are found in Section 4.3. Section 4.4 covers the chemical analyses, including T_g, thermal mechanical and photoacoustic IR analyses. Section 4.5 summarizes the earlier subsections. More detailed results have been placed in Appendix C. Raw data are located in Appendices E and F.

Throughout this report, specific results are provided for use in determining whether a particular cleaning solution is acceptable for a particular application in consideration of the adhesive(s) present and the operational ramifications of changes in hardness and T_g or water absorption. (For example, see Tables 13, 15 and 19 in the main body and Tables C-3 and C-4 in Appendix C.)

4.1.1 Responses

The most important response from the lap shear experiment was the amount of force required to separate the coupons. The failure type was also analyzed. This measure was captured in terms of percentage of the bonded area experiencing adhesive failure. Results presented for bulk samples include physical data—dimensions, percentage water absorption, and hardness—and chemical analysis results, specifically apparent glass-transition temperature (T_g). Chemical thermal analysis and photoacoustic IR analysis were also performed.

4.1.2 Analysis

Statistical analysis was performed with the two data sets, lap-shear and bulk. These data are presented in Appendix E and Appendix F, respectively. Both high-level and detailed analyses were performed on each response. Analyses investigated the effect of each independent variable by itself: adhesive, detergent, cleaning method, temperature, and group. Combinations of variables were also investigated, as were interactions, using multiple regression. The temperature was treated both as a discrete variable and as a continuous variable. Sonication frequency was also studied alone and in combination with other factors.

Extensive exploration and analysis of the data was performed. This involved correlation analysis, graphical analysis, calculation of means and standard deviations, and multivariate regression analysis. Both main effects and interactions were explored. A main effect is a change in a response value as a function of an independent variable, such change being unrelated to the values of other variables. Alternatively, an interaction effect is a change in the effect of one variable where the magnitude of that change is dependent upon the value of another variable. The design of this experiment was primarily directed toward determination of main effects, but conclusions were possible concerning certain interactions.

Data tabulation, validation, and initial manipulation were performed using Quattro Pro (Version 4.0 for DOS) on IBM or IBM-compatible personal computers. The multiple regression and other statistical analyses were performed using Minitab Statistical Software, Release 7.1, on Battelle's VaxCluster.

Analysis of rich data sets such as these is an iterative, evolutionary process. Each analysis step provides insights and suggests additional analyses. Some of these results will be discussed in detail in the subsections that follow.

4.2 Lap Shear Results

4.2.1 Lap Shear Responses

The four responses collected on lap shear samples are the following:

- Force required to separate the coupons of a lap-shear specimen after a single simulated cleaning exposure (S-PSI)
- Force required to separate the coupons of a lap-shear specimen after 12 simulated cleaning exposures (L-PSI)
- Percentage of bond area experiencing adhesive failure after a single exposure (S%Adh)
- Percent of bond area experiencing adhesive failure after 12 exposures (L%Adh).

The more general results of the analyses performed on these responses are presented in the subsections that follow. Detailed analysis is described in Appendix C, Section *C.1*.

4.2.2 Group Effect

The first analysis performed was for the effect of group. The experiment was designed in two blocks to control for the effects of learning, batch-to-batch differences in mixing, etc. It was hoped that there would be no significant difference of results between groups, but it was predicted that such a difference was possible. Table 10 indicates which adhesives were run in each group. For further detail, please reference Appendix C.

Table 10. Adhesives by Group

Group	Adhesive									
	1	2	3	4	5	6	7	8	9	10
1	✓	✓					✓			
2						✓			✓	✓
3			✓	✓				✓		
4		✓			✓				✓	
5	✓		✓				✓			
6			✓	✓	✓					✓

It was determined that specimens in Group 1 required significantly less force to break following single exposure and 12 exposures. Group 3 also shows a significantly lower force to break after single exposure, as does Group 5. The results for Group 1 could be explained based on learning and equipment adjustment issues. This might also be the explanation for the Group 3 single-exposure result. The variation noted in Group 5 cannot be accounted for.

For all responses, significant block and group effects emerged from the regression analyses. The higher variability in Block I could be caused by procedural issues, but it could also be due to the materials present. Elimination of two sealants, PR1422B and GE RTV60, after Block I and inclusion of very few sealant trials in Block II could explain the differences. The significant effects found among groups is consistent with the adhesive differences. Thus, the conclusion is that the experimental procedure as executed was acceptable and there was no adverse effect of learning, mixing, or other procedural factors.

4.2.3 Failure Type

Table 11 presents the average percentage of bond area experiencing adhesive failure for each adhesive. Appendix B contains the protocol used in assigning the type of bond failure observed in the lap shear samples. The predominant failure mode is noted next to each percentage, in accordance with the failure mode definitions in Section 4.2.1. The sealants, Ablestik 724/14C and GE RTV60, experienced adhesive failure. In fact, the GE RTV60 samples all separated prior to being pulled. LCA9/BA5 also exhibited adhesive

failure predominantly. This adhesive proved to be the weakest of the epoxies. All other adhesives tested experienced delamination as the primary failure mode. Failure of the other epoxies was associated with delamination rather than loss of interfacial attraction. Bond failure did not change throughout the study for a given adhesive. Therefore, failure type is not a usable indicator of degradation.

Table 11. Failure Mode by Adhesive

	Single Exposure		12 Exposures	
Adhesive	Adhesive Failure (%)	Failure Mode	Adhesive Failure (%)	Failure Mode
1. FA8/BA5	86	Delamination	84	Delamination
2. LCA9/BA5	97	Adhesive	97	Adhesive
3. LCA4/BA5	70	Delamination	72	Delamination
4. LCA4XM/BA5XM	70	Delamination	73	Delamination
5. ABLESTIK 724/14C	93	Adhesive	100	Adhesive
6. PR 1422B	77	Delamination	62	Delamination
7. C-7/Activator W	73	Delamination	69	Delamination
8. GE RTV60	100*	Adhesive	100*	Adhesive
9. Epon 828/Versamid 125	70	Delamination	71	Delamination
10. Eccobond 2216	65	Delamination	69	Delamination

* Coupons separated prior to immersion

4.2.4 Force to Separate Coupons

A detailed analysis of the lap-shear data was performed on the two responses, S-PSI and L-PSI. Table 12 presents the average force required for each adhesive for a single exposure and for 12 exposures. It is seen that the sealants, Adhesives 5 (Ablestik 724/14C), 6 (PR1422B), and 8 (GE RTV60), have noticeably less bonding strength than the others, which are all epoxies.

Table 12. Average Force by Adhesive

Adhesive	Force to Pull Lap Shears	
	Single Exposure (PSI)	12 Exposures (PSI)
1. FA8/BA5	2,401	2,471
2. LCA9/BA5	2,035	2,122
3. LCA4/BA5	2,626	2,640
4. LCA4XM/BA5XM	2,584	2,629
5. ABLESTIK 724/14C	1,173	853
6. PR 1422B	120	129
7. C-7/Activator W	3,388	3,340
8. GE RTV60	0*	0*
9. Epon 828/Versamid 125	3,545	3,507
10. Eccobond 2216	3,006	3,042
All	2,818	2,796

* Coupons separated prior to immersion

The difference between the strength of the bond after 12 exposures when compared to the strength of the bond after a single exposure was analyzed in several ways, and every analysis produced the same result—that the change in strength of the bond is completely random. There is no effect of repeated exposures that is discernible from this data.

The regression analyses for both L-PSI and S-PSI produced results that accounted for nearly 90 percent of the variability in the data. This is a very good result for laboratory experimentation of this type.

In the analysis of detergents, only distilled water and 1,1,1-trichloroethane showed significant differences from the unexposed controls. Samples exposed to either of the solutions showed significantly less strength than the unexposed controls. Detergent 2 (Brulin 815GD) and Freon 113 correlated with weaker bonds. No other detergent showed significant degradation of the strength of the bond, including the acidified and alkaline-adjusted water control. Following a single exposure, there was no effect of detergent that proved significant when compared to the unexposed control. The adhesive differences noted in the previous paragraph for life-cycle testing also manifested themselves after a single exposure.

No effect of cleaning method was found for either life-cycle or single-exposure testing.

Elevating the temperature had an effect on the strength of the bond after life-cycle exposure. Both 135 F and 190 F showed significant degradation. For the single exposure case, there was no significant effect of the middle temperature, but the highest temperature used showed a significant degradation in the bond.

4.2.5 <u>Comparison with Other Detergent Controls</u>

The lap shear data was analyzed to compare the results individually with each of the control solutions, in contrast to the previous comparisons that were done with the unexposed condition as the control. These results have been synthesized with results from a similar analysis of bulk data and are presented with the summary of results.

4.3 <u>Bulk Sample Results</u>

This section summarizes the results of the analysis of the bulk sample data. The raw data have been provided to AGMC on diskette. The response values used in the analysis are presented in Appendix F. SSH is Shore hardness after a single exposure, LSH is Shore hardness after 12 exposures. Shore A was used for the sealants and Shore D for the epoxies. SdV is change in volume after a single exposure, LdV is change in volume after 12 exposures, SdWt is change in sample weight after a single exposure, and LdWt is change in sample weight after 12 exposures. The change values for volume and weight are expressed as percentages of the initial volume or weight. Table 13 presents summary statistics for hardness (Shore A or Shore D), change in volume, and change in weight after a single exposure and after 12 exposures. These findings are discussed in the subsections that follow. More detailed results are presented in Section *C.2* of Appendix C.

Table 13. Bulk Sample Data—Statistics

SSH Statistics			
Adh	Avg	StDev	n
1	81.94	5.32	30
2	91.12	4.53	33
3	87.30	2.61	58
4	86.36	2.51	28
5	84.87	2.27	24
6	16.94	4.32	8
7	74.75	3.03	34
8	29.05	3.37	16
9	77.65	2.97	28
10	63.84	4.36	28
All	76.96	17.90	287

LSH Statistics			
Adh	Avg	StDev	n
1	81.71	3.14	26
2	92.29	3.71	28
3	86.39	1.67	58
4	85.15	2.31	28
5	86.38	1.59	24
6	20.95	7.17	8
7	73.34	3.33	28
8	34.13	6.22	16
9	74.74	3.63	28
10	63.38	7.20	28
All	76.51	17.22	272

SdV Statistics			
Adh	Avg	StDev	n
1	-0.98	2.05	21
2	-1.91	4.92	23
3	-0.13	2.56	58
4	0.28	1.16	28
5	0.56	2.51	24
6	*	*	0
7	0.30	0.67	26
8	-0.56	0.93	14
9	0.18	6.20	26
10	0.32	2.15	24
All	-0.16	3.13	244

LdV Statistics			
Adh	Avg	StDev	n
1	0.16	1.12	26
2	-0.09	0.87	28
3	0.61	2.80	58
4	0.99	1.43	28
5	5.41	4.71	24
6	-0.04	14.26	7
7	0.69	0.98	29
8	-2.66	1.95	16
9	2.25	3.95	28
10	3.24	3.92	28
All	1.20	3.90	272

SdWt Statistics			
Adh	Avg	StDev	n
1	-0.14	1.10	31
2	0.02	0.06	33
3	0.03	0.07	56
4	0.28	0.06	28
5	0.61	0.91	24
6	-1.82	1.56	7
7	0.23	0.31	34
8	6.03	10.98	16
9	0.37	0.36	27
10	-0.11	1.67	26
All	0.40	3.00	282

LdWt Statistics			
Adh	Avg	StDev	n
1	0.22	1.14	26
2	0.16	0.16	28
3	0.12	0.10	58
4	-0.09	0.59	28
5	1.28	2.42	24
6	-6.15	1.67	8
7	1.22	1.19	28
8	5.80	10.71	16
9	1.46	1.31	28
10	0.50	1.90	28
All	0.66	3.32	272

4.3.1 Percentage Water Absorption

In general, small changes were observed in the analysis of the epoxy data related to percent weight gain after single and life exposures. The measure of interest was percentage change in weight based on the initial weight of the sample. Samples after simulated life cycle showed only a slightly greater increase in weight compared to those after a single exposure, and only in isolated cases was the change sufficiently large to be considered a problem.

Statistics for water absorption are also presented in Table 13. The percentage change in weight over the life cycle (LdWt) ranged from 8 percent decrease to 33 percent increase.

The difference between change in weight after a single exposure and after the simulated life cycle was not great. Rarely was it more than 5 percent. There is a correlation of 0.846 between change in weight after first exposure and change in weight after 12 exposures, both changes being calculated with respect to initial weight prior to exposure.

4.3.2 Dimensional Changes

The analysis for dimensional changes was performed on volume after preliminary assessment of the data indicated that changes in any single dimension were typically small. Dimensional changes showed greater variation than weight changes, but still fell within acceptable limits for most adhesives evaluated. For all samples, the average change in volume was a decrease of 0.16 percent after a single exposure and an increase of 1.2 percent over the life cycle. Values ranged from a decrease of 18.8 percent to an increase of 18.2 percent after a single exposure (SdV). The range of change in volume after 12 exposures (LdV) was from a decrease of 16.5 percent to an increase of 28.2 percent. Statistics on dimensional changes are presented in Table 13.

For sealants, there was a high variability in dV, partly due to the amount of distortion of the samples and associated difficulty in accurately calculating volume. Still, this distortion indicates that most cleaning processes adversely affect these adhesives. Adhesive 6

(PR1422B) did not lend itself to volumetric analysis because of severe distortion of the samples after exposure.

The group effect was again noted, but again it was attributable to the combination of adhesives rather than to procedural factors. When the method was sonication, there was less of an increase in volume. The effect was present and similar in magnitude at both frequencies of sonication. This could indeed be due to the method of cleaning or it could simply be due to the shorter duration of exposure under the sonication procedure (5 minutes versus 60 minutes per exposure for soaking).

Results for specific adhesive-detergent combinations are presented in Appendix C, Section *C.2.2.*

4.3.3 Hardness

Shore hardness values averages for each adhesive are presented in Table 13. Inspection of the hardness data revealed no significant change in hardness between single exposure and simulated life cycle. The sealants (Adhesives 5 [Ablestik 724/14C], 6 [PR1422B], and 8 [Epon 828/Versamid 125]) actually exhibited a tendency to increase hardness slightly over the life cycle which might result in the loss of flexibility, an important sealant characteristic.

Some of the detergents were associated with significantly greater hardness than the unexposed control condition (notably, Versaclean). Only PF Degreaser and alkaline-stabilized water were associated with significantly decreased hardness values. No significant effects of method or temperature were noted.

Shore hardness values for particular adhesive-detergent combinations may be found in Appendix C, Section *C.2.3.*

4.4 Chemical Analysis

4.4.1 T_g Analysis

Thermal analysis was performed on bulk specimens to determine the glass-transition temperature (T_g) for most of the trials following exposure for the simulated life cycle. In addition, measurement of T_g was performed after a single exposure for most of the trials in Block I. The average values obtained for each adhesive are presented in Table 14.

Table 14. T_g Results by Adhesive

Adhesive	Single Exposure		Life-Cycle Exposure	
	T_g (°C)	Std. Dev.	T_g (°C)	Std. Dev.
FA8/BA5 (1)	58.01	5.71	89.96	15.38
LCA9/BA5 (2)	54.20	3.07	83.05	13.16
LCA4/BA5 (3)	no data		82.33	10.73
LCA4XM/BA5XM (4)	61.94	14.19	78.44	7.69
Ablestik 724/14C (5)	no data		-25.27	5.63
Adhesive C-7/Activator W (7)	50.57	1.66	59.25	9.03
Epon 828/Versamid 125 (9)	59.28	3.57	57.76	8.84
Eccobond 2216 (10)	no data		18.57	3.81

T_g is a sensitive indicator of changes in the adhesives and serves as an early warning indicator of potential future problems. Increasing T_g values, as found in this study, are not detrimental, and embrittlement was not observed. Decreases in T_g found are indicative of softening of the adhesive which can cause detrimental changes in performance.

An increase in T_g for the epoxies was associated with all methods, while a significant decrease in T_g of the elastomer Ablestik 724/14C occurred when the method was soaking. Thus, the change could be due to the soaking as a cleaning method or to the fact that soaking entails much longer exposure to the cleaner than does sonication.

There was a tendency for T_g to increase over the life cycle as samples continued to cure. For the unexposed controls, this increase averaged 6%. It was 20% for

exposed samples at room temperature and at 190 F, and the increase was 30% at 135 F. The significance of this middle-temperature effect remains to be interpreted.

One conclusion drawn from the T_g analysis is that detergents, especially EZE 240 and Intex 8125, affect epoxies differentially. This conclusion is based on the higher variance of life-cycle data versus single-exposure data. Another conclusion is that other effects, such as airflow time and other factors outside the design, affect T_g and account for some of the observed differences. A third conclusion is that 1,1,1-trichloroethane and Freon 113 do not, in general, affect epoxies while they do adversely affect elastomers like Ablestik 724/14C.

Regression analysis indicated the factors that appeared significant after life-cycle testing were not apparent following a single exposure. This indicates that a single exposure is not sufficient to determine life-cycle effects.

4.4.2 Thermal Mechanical Analysis

Listed in Table 15 are the individual trials showing significant degradation in their percent weight change, lap shear, or visual characteristics. These trials were chemically analyzed selectively to determine if analysis would corroborate the observed degradation. These trials also represent the largest observed changes in percent weight change and lap shear.

Further thermal analysis of selected samples was conducted using a Perkin Elmer System 7 TMA instrument operated in the expansion mode. Analysis was performed on the bulk sample coupons. A summary of the results of the samples analyzed can be found in Table 16. Ablestik 724/14C and PR 1422B, both sealant-type materials, were dimensionally stable up to their sharp melting points of 190 C/374 F and 230 c/446 F respectively. No further analysis efforts were expended on these materials due to their melting behavior except to note that T_g of Ablestik 724/14C increased significantly when exposed to Oakite Liquid Detergent #2 (T_g = -16.4 C) or distilled water (T_g = -11.8 C). Otherwise, T_g remained in the range (-30.0 to -24.1) for this solvent. In general the other adhesive materials expanded over the temperature range investigated, 0-230 C.

Table 15. Candidates for Chemical Analysis

Trial #	Weight	Lap Shear	Visual	Criteria for Selection
3	✓	✓	✓	wt - <300% of control
15	✓			unexposed
17	✓			
18	✓			lap shear - ≤90% of unexposed
20	✓			control
35	✓			
53	✓			
61	✓	✓		
63		✓		
69	✓			
67		✓		
71	✓			
78		✓		
80	✓	✓	✓	
83		✓	✓	
90	✓		✓	
92			✓	
93	✓		✓	
94	✓	✓	✓	
95	✓	✓	✓	
100	✓			
103	✓	✓	✓	
104	✓	✓	✓	
105	✓	✓	✓	
110		✓	✓	
111	✓	✓		
113	✓		✓	C4A
115			✓	
118			✓	DI H_2O
119			✓	
120			✓	
124		✓		

Table 16. TMA Results—Degraded Samples

Adhesive	Trial Number	Percent Expansion/ C Range	Comments
1. FA8/BA5	Control-A	3.95/75-230	Linear expansion after 75 C
1. FA8/BA5	15	2.62/85-230	Linear expansion after 85 C
2. LCA9/BA5	Control-A	1.56/0-230	Very little difference, expansion is linear
2. LCA9/BA5	3	1.56/2.10/0-300	Very little difference, expansion is linear
3. LCA4/BA5	Control-C	2.13/88-230	Linear expansion after 88 C
3. LCA4/BA5	120	2.31/83-230	Linear expansion after 83.1 C
4. LCA4XM/BA5XM	Control-C	2.0/71.2/230	Linear expansion after 71.2 C
4. LCA4XM/BA5XM	53	1.6%/91.9-230	Linear expansion after 91.9 C
5. Ablestik 724/14C	111	0.0/25-190	No changes until 190 C/374 F, then melts sharply
6. PR1422B	Control-B	0.0/25-230	No changes until 230.1 C/446 F, then melts rapidly
7. Adhesive C-7/ Activator W	Control-A	3.3/60.7-230	Inflection point at 60.7 C, then expands linearly
7. Adhesive C-7/ Activator W	17	3.16/0-230	No inflection point, linear expansion over range
8. GE RTV60	Control-C	6.8/0-230	Constant linear expansion over range, large change
9. Epon 828/Versamid 125	Control-B	4.13/0-230	Very little difference between samples, linear expansion over range
9. Epon 828/Versamid 125	80		
10. Eccobond 2216	Control-B	3.3/0-230	Very little difference between samples, linear/expansion over range
10. Eccobond 2216	104		

Adhesives 2 (LCA9/BA5), 9 (Epon 828/Versamid 125), and 10 (Eccobond 2216) did not show any differences between the unexposed control and the selected degraded trial. All three of these adhesives showed a linear expansion ranging from 1.5 to 4.1 percent starting at 0 C to the 230 C maximum temperature scanned. Adhesives 1 (FA8/BA5) and 4 (LCA4XM/BA5XM) showed a higher temperature of expansion onset than the unexposed control while Adhesives 3 (LCA4/BA5) and 7 (Adhesive C-7/Activator W) showed the opposite trend.

In general, TMA results were not helpful in supporting observed degradation. Earlier, it was felt that extensive development of baseline performance of the adhesives would not be required and that changes in the exposed samples would be large and easily identifiable. Experimentally this was found not to be the case, with only slight changes observed. Therefore, the best use of the TMA data is to establish the basic performance of the adhesives for use in future studies.

4.4.3 Photoacoustic IR Analysis

Photoacoustic Fourier transform infrared (PA FT-IR) spectroscopy has become a popular technique for the analysis of polymeric coatings and adhesives. In contrast to transmission FT-IR experiments, PA FT-IR measures absorption of the infrared radiation thus eliminating the problem of detector saturation caused by strong absorption bands in the sample. PA FT-IR also has the advantage of requiring little or no sample preparation.

4.4.3.1 Summary of PA FT-IR Results

The bulk adhesive and lap shear samples were analyzed using photoacoustic FT-IR spectroscopy in an attempt to identify chemically related reasons for the adhesive failure. All data descriptions and interpretation are with respect to a subtraction spectrum generated by subtracting the bulk adhesive unexposed spectrum from the cleaned adhesive spectrum, in the case of the plaques. Figure 5 illustrates this data for the bulk adhesive of Trial 103 with the top spectrum being the cleaned plaque, the middle spectrum being the unexposed plaque, and the bottom spectrum being the difference of the two. In the difference or subtraction spectrum, positive peaks indicate that the exposed material has more of or a new functional group as compared to the unexposed material. When the difference spectrum has a negative band, the unexposed adhesive has more of a certain functional group than does the exposed material. With regard to the lap shear samples the subtraction spectrum was generated by subtracting the bulk adhesive spectrum from the lap shear spectrum. Figure 6 represents the spectra for the lap shear sample L104-B. The top spectrum is the exposed lap shear, the second spectrum is the detergent for comparison

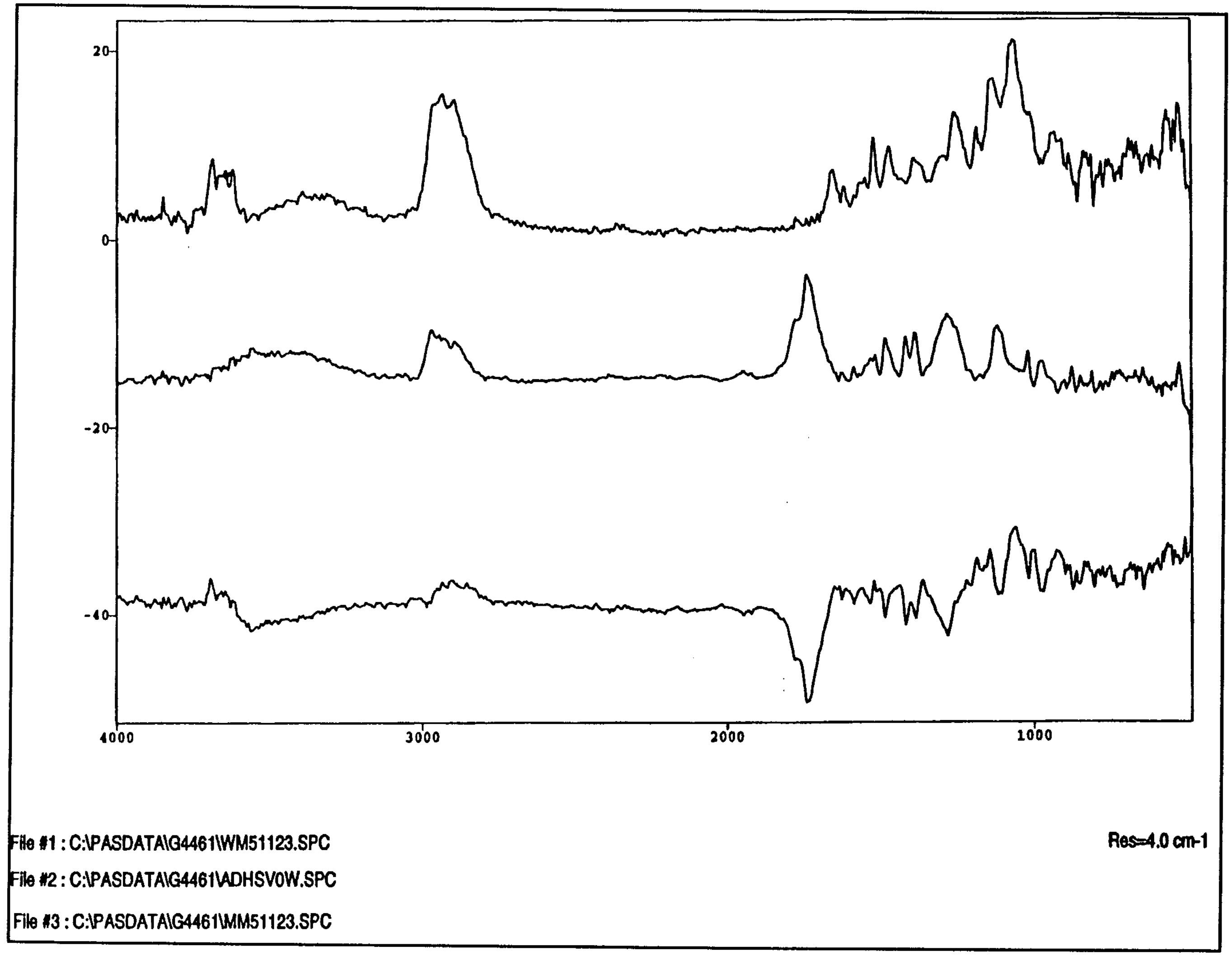


Figure 5. Example of PA-FTIR Spectrums, Trial 103

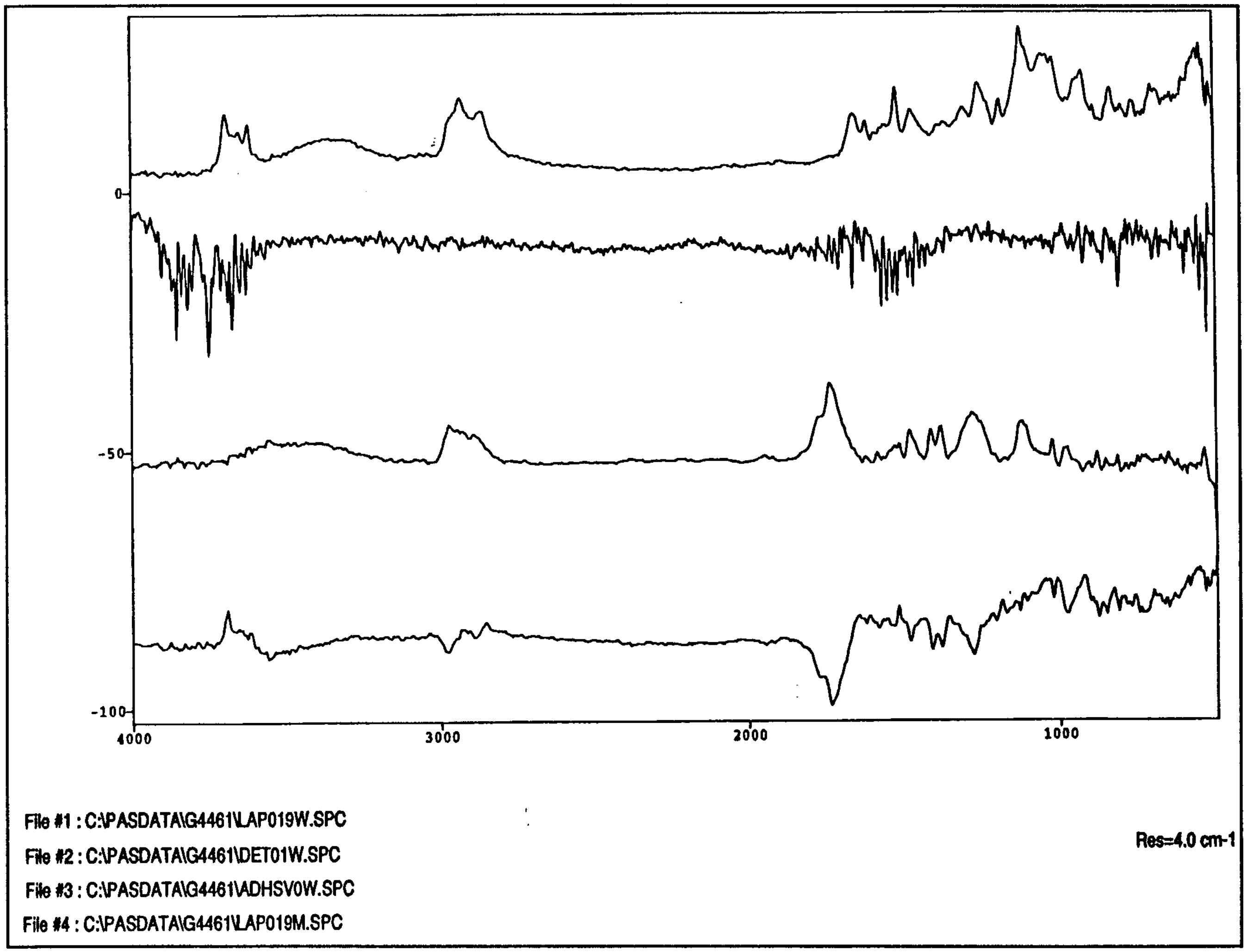


Figure 6. Example of PA-FTIR Spectrums, Lap Shear Samples L104-B

purposes, the third spectrum is the unexposed adhesive, and the fourth spectrum is the difference between the exposed and unexposed spectra. Positive and negative bands mean the same as described above. During the analysis the respective detergent spectrum was also considered in order to ascertain when and if the detergent or the cleaning process was having an effect on the adhesive materials.

4.4.3.2 Bulk-Sample Data

It appears that Adhesives 10 (Eccobond 2216) and 1 (FA8/BA5) are being attacked by the cleaning process and/or the detergent since there is loss of carbonyl group, pick up of water, and, in some cases, pick up of detergent. Adhesive 3 (LCA4/BA5) also appears to be attacked by the cleaning process and/or detergent since new bands are formed in some cases and there are increased interactions by some of these new bands. In contrast, Adhesives 2 (LCA9/BA5) and 5 (Ablestik 724/14C) do not appear to be adversely affected by the detergent and cleaning bath. Adhesives 7 (Adhesive C-7/Activator W) and 9 (Epon 282/Versamid 125) have minor changes occurring in them suggesting hydrolysis, but not enough to conclusively analyze the data.

4.4.3.3 Lap Shear Data

All of the unexposed adhesives in the lap shear studies had "new" infrared vibrational modes present suggesting interaction or reaction with the aluminum substrate. However, the results observed with the exposed lap shear measurements remained consistent with the bulk-sample data. Adhesives 10 (Eccobond 2216) and 3 (LCA4/BA5) appear to be affected by the detergent and cleaning process. Adhesive 1 (FA8/BA5) had few changes, while Adhesive 2 (LCA9/BA5) flaked from the substrate and showed apparent damage. Adhesives 4 (LCA4XM/BA5XM), 5 (Ablestik 724/14C), and 9 (Epon 828/Versamid 125) had few, if any, changes suggesting that the cleaning process had few changes in the lap shear samples. In contrast, Adhesive 6 (PR1422B) had obvious carboxylic acid formation in the lap shear sample but not in the bulk adhesive. This is interpreted as being negative at this time; however, the acid could be a by-product of the crosslinking reaction and not pose a real threat to the interfacial integrity of the sample. Adhesive 7 (Adhesive C-7/Activator W)

has the disappearance of a band after being exposed to the cleaning solutions. The disappearance of this band is interpreted as being negative since it suggests that the adhesive is being degraded.

4.5 Summary of Results

This section summarizes the many findings described in the prior subsections.

4.5.1 Comparison of Detergent Controls

Table 17 summarizes the differences in the performance measures for each of the detergent controls run in the study versus a statistical composite of the adhesives. For each response (strength of bond, change in weight, change in volume, Shore hardness, and T_g), a series of computer runs was made using a different detergent as the control. These five detergent controls are listed at the left of Table 17. The entries in the cells of a row describe how the control at the top of the column compared with the solution noted at the left.

In general, there were few differences. The following discussion is based on a comparison to the statistical combination of all the adhesives. The nonaqueous controls, 1,1,1-trichloroethane and Freon 113, behaved similarly, when compared to the other controls. Bond strength differences were in the direction of weaker bonds with the organic solvents. Where differences in weight appeared, the tendency was for an increase in weight with the organic controls compared to the aqueous controls or no exposure. There were only minor differences between the control solutions and the unexposed control condition. A stronger bond occurred with both pH-adjusted water solutions, but not with neutral distilled water. The only statistically significant (at the 95% level) difference in bond strength was in the comparison of pH-adjusted water with the organic controls. The other bond strength differences noted are significant at the 80% level. No differences in T_g were noted that were significant at the 95% level. Those differences noted as "slight" increases or decreases would be significant at the 80% level.

Table 17. Comparison of Detergent Controls at Top with Control Basis at Left*

Control Basis for Analysis	Detergent Controls					
	No Exposure (Control)	Distilled Water	1,1,1-Trichloroethane	Freon 113	Acidified Distilled Water	Alkaline-Adjusted Distilled Water
Distilled Water	- No diff. in bond - No diff. wt - No diff. volume - No diff. Shore - No diff. in T_g	⊗	- No diff. in bond - 2.5% incr. in wt - No diff. volume - No diff. Shore D - No diff. in T_g	- No diff. in bond - 3% incr. in wt - No diff. volume - No diff. Shore D - No diff. in T_g	- 10% stronger bond - No diff. wt - No diff. volume - No diff. Shore D - Slight decr. T_g	- 12% stronger bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g
1,1,1-Trichloroethane	- No diff. bond - 3% decr. in wt - No diff. volume - No diff. Shore D - No diff. in T_g	- No diff. in bond - 2.5% decr. in wt - No diff. volume - No diff. Shore D - No diff. in T_g	⊗	- No diff. in bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g	- 13% stronger bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g	- 13% strngr bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g
Freon 113	- 8% stronger bond - 3% decr. in wt - No diff. volume - No diff. Shore D - No diff. in T_g	- No diff. in bond - 3% decr. in wt - No diff. volume - No diff. Shore D - No diff. in T_g	- No diff. in bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g	⊗	- 15% stronger bond - 2% decr. in wt - No diff. volume - No diff. Shore D - No diff. in T_g	- 16% strngr bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g
Acidified Distilled Water	- No diff. in bond - No diff. wt - No diff. volume - No diff. in Shore D - Slight incr. T_g	- 10% weaker bond - No diff. wt - No diff. volume - No diff. Shore D - Slight incr. T_g	- 13% weaker bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g	- 15% weakr bond - 2% incr. in wt - No diff. volume - No diff. Shore D - No diff. in T_g	⊗	- No diff. in bond - No diff. wt - No diff. volume - No diff. Shore D - Slight incr. T_g
Alkaline-Adjusted Distilled Water	- No diff. in bond - No diff. in wt - No diff. volume - Decreased Shore D - No diff. in T_g	- 12% weaker bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g	- 13% weaker bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g	- 16% weaker bond - No diff. wt - No diff. volume - No diff. Shore D - No diff. in T_g	- No diff. in bond - No diff. wt - No diff. volume - No diff. Shore D - Slight decr. T_g	⊗

* Matrix comparisons refer to detergent controls over control basis (i.e., read matrix from top to bottom, right to left). For example, the second shaded column's second cell indicates that the distilled water samples weighed 2.5% less than the samples exposed to 1,1,1-trichloroethane.

4.5.2 Results for Adhesive-Detergent Combinations

In-depth analysis was performed on the data for all 12 responses for each of the 120 combinations of adhesives and detergents (including controls). Table 18 presents the results for T_g and the epoxies. A plus sign in the cell indicates that an increase in T_g was observed and the combination may be considered unless the application renders an increase in T_g unacceptable. Similarly, no entry in the cell implies that the T_g analysis provided no reason to rule out the combination. No recommendation can be made for the cells denoted "NE", because these cases were not evaluated. The minus sign indicates that a decrease in T_g was observed and the combination should be avoided. This conclusion is based on the assumption that a decrease in T_g is unacceptable for most applications.

Table 18. Recommendations about Epoxy-Detergent Combinations with Regard to Changing T_g

Detergent	Epoxy						
	FA8/BA5 (1)	LCA9/BA5 (2)	LCA4/BA5 (3)	LCA4XM/ BA5SM (4)	C-7/Activator W (7)	Epon 828/ Versamid 125 (9)	Eccobond 2216 (10)
Versaclean	+	+		NE	-	+	-
Brulin 815 GD	NE	+	-	+	-	-	
EZE 240	+	+	-	+	-	-	NE
Intex 8125	+	+	-	NE	-	-	
MSI 1025	NE	+	-	+		-	NE
Oakite Liq. Det. #2	+	+	-	NE		-	NE
PF Degreaser	NE	+	-	NE	-		NE
Citranox	+	+		+	-	-	NE
Water	NE	+		+		-	NE
1,1,1-Tri-chloroethane	+	+	-	NE	-		
Freon 113	NE	+	-	NE	+	-	

NE = Combination not evaluated.
No Entry = No significant change in T_g noted over life cycle = acceptable combination.
\+ = Significant increase in T_g, but acceptable combination unless application indicates that an increase in T_g is unacceptable.
\- = Significant decrease in T_g (>15%) = combination not recommended.

Table 19 presents a synthesis of the findings for all adhesive-detergent combinations evaluated for all responses tested. Note that every combination was not run with every method and temperature; so these effects of process may be embedded in these results. Only further experimentation can support or refute this concern. However, with this understanding of the power of the screening design upon which this study was based, the reader may use this table to make decisions about the potential acceptability of specific combinations for particular applications.

If the combination was not evaluated at all, the entry "NE" occupies the cell and no conclusions can be drawn. The entry "NE-T_g" indicates that thermal analysis was not performed, etc. Where there is no NE entry and no comment about a particular response, the combination was no different than the unexposed control. Differences of interest are noted below.

- degradation in bond strength $>12\%$
- increase in bond strength $\geq 10\%$
- decrease in T_g $>5\%$
- weight gain $>1.3\%$
- statistically significant volume change

An X in the lower right corner of the cell indicates a recommendation against the use of this combination. Such a recommendation is made if the degradation in bond strength exceeded 20% or if T_g decreased more than 25%. Softening, physical distortion, and solvent absorption were also grounds for rejection, though the criteria are less rigorous and more application specific. A question mark recommends caution against the combination unless there are compelling reasons to use it and further tests are run to confirm its acceptability for the intended application. Typically caution is advised if degradation in bond strength is of the order of 15-20%.

Table 19. Summary of Results for Adhesive-Detergent Combinations for All Responses

Detergent	Epoxy: FA8/BA5	Epoxy: LCA9/BA5	Epoxy: LCA4/BA5	Epoxy: LCA4XM/BA5XM	Epoxy: C-7/Activator W	Epoxy: Epon 828/ Versamid 125	Epoxy: Eccobond 2216	Sealant: Ablestik 724/14C	Sealant: PR1422B	Sealant: GE RTV60
Versaclean	15% weaker after 1 exp; 10% weaker over life cycle		1.0% increase in volume	2.6% increase in volume; 0.5% increase in wt; NE-T_g	T_g decreased 7%	T_g increased 24%	Softened immediately; T_g decreased 34%; 3% inc in wt *X*		*X*	*X*
Brulin 815 GD	NE-T_g	Bond weakens 17% initially but ok over life cycle ?	NE-Lap Shear; T_g decreased 17%		25% stronger after first exp but 20% weaker over life; T_g decreased 12% *X*	Softened immediately; T_g decreased 29%; bond weakens; 4% increase in wt *X*	16% stronger bond		*X*	*X*
EZE 240	Bond weakens 22% *X*		T_g decreased 25%		T_g decreased 12%	Bond weakens 25% initially but OK over life; T_g decreased 21% ?	Softened immed, but OK over life; 10% stronger bond; NE-T_g		*X*	NE-T_g. *X*
Intex 8125	Bond weakens 13% initially and same over life cycle		Bond weakens 12%; T_g decreased 16%	4.5% increase in volume; NE-T_g	Bond weakens 2.3%; 4% incr in wt; 2.5% incr in volume; T_g decreased 60% *X*	Bond weakens 22%; T_g decreased 21% *X*	Softened over life; bond weakens 14% *X*	10.8% increase in volume. *X*	4% incr in wt *X*	*X*
MSI 1025	Softens 5% immed but OK over life; 30% increase in strength of bond; NE-T_g.	Bond weakens 13%	T_g decreased 17%	1.3% increase in wt		Bond weakens 15% initially but OK over life; T_g decreased 6% ?	20% stronger bond; NE-T_g		*X*	NE-T_g. *X*
Oakite Liq Det #2	Bond weakens 30% *X*	Bond weakens 20% *X*	NE-Lap Shear; T_g decreased 27% *X*	1.3% increase in wt; NE-T_g	11% stronger bond	Bond weakens 16%; T_g decreased 10%	17% stronger bond; NE-T_g	9.5% increase in volume. *X*	Softened immed. *X*	NE-T_g. *X*
PF Degreaser	NE	Bond weakens 15% ?	T_g decreased 19%	NE-Lap Shear	T_g decreased 9%		NE	9.8% increase in volume. *X*	*X*	Softens excessively. 13% incr in wt. NE-Lap Shear, T_g. *X*
Citranox			NE-Lap Shear		T_g decreased 7%	Softened immed but OK over life; 4% increase in wt; T_g decreased 20% *X*	33% stronger bond; NE-T_g		*X*	NE-T_g, Shore OK. *X*
Water	Softens immed but OK over life; NE-T_g				Bond weakens 12%	Bond weakens 12%; T_g decreased 20% ?	Bond 10% weaker; NE-T_g		Softened. *X*	NE-T_g. *X*
1,1,1-Trichloroethane		Bond weakens 20% *X*	T_g decreased 40%	Softens immed; NE-Lap Shear, T_g *X*	10% stronger bond	Softened over life; 2% incr in wt *X*	Softened; 3% incr in wt *X*	9% increase in wt. *X*	*X*	Softens initially but OK over life. 5% incr in wt. *X*
Freon 113	NE-T_g		NE-Lap Shear; T_g decreased 17%	NE-T_g	Bond weakens 28% *X*	T_g decreased 17% ?	20% stronger bond; 4% decrease in wt	10% increase in volume *X*	Softened over life. *X*	28% increase in wt immed and over life. *X*
General Comments about Adhesive Performance after Exposure	No sig change in wt or volume. Chem anal showed chem change and pickup of detergent. T_g increased.	No softening. T_g increased. Adhesive failure on lap shears. Weakest of the epoxies. No sig change in wt or vol. No chem changes.	No softening; no sig degradation in strength of bond; no appreciable dimensional changes; no chem changes.	Unexp sample softened over life cycle-others did not; wt decreases; no volume changes except 4; T_g increased; no chem changes	No softening; very strong bond except as noted; increase in volume with alkaline water; minor chemical changes-hydrolysis	Strongest bond except as noted; wt increases; volume decreases; minor chem changes-hydrolysis	Softer than other epoxies; maintained SH over life except as noted; strong bond except as noted; weight increases	No change in SH. Adhesive failure on lap shear. Decreases in weight. Significant distortion. No chem changes.	Unexp control hardened over life; adhesive failure; softened and distorted considerably; decreased in wt; some chem changes	Adhesive failure. Gains wt with non-aqueous det. Distorts. Should not be washed.

Legend For Table 19

NE = Combination not evaluated.
NE-T_g = T_g analysis not performed on combination.
NE-Lap Shear = Lap shear analysis not performed on combination.
NE-Bulk = Bulk sample analysis not performed on combination.
***X* = Do not use! Degradation observed is statistically significant at 95% level.**
? = Do not use this combination without application-specific testing. Caution advised because of the degradation observed which was significant at the 80% level.

Table 19. Summary of Results for Adhesive-De

Detergent	Epoxy				
	FA8/BA5	LCA9/BA5	LCA4/BA5	LCA4XM/BA5XM	C-7/Activator W
Versaclean	15% weaker after 1 exp; 10% weaker over life cycle		1.0% increase in volume	2.6% increase in volume; 0.5% increase in wt; NE-T_g	T_g decreased 7%
Brulin 815 GD	NE-T_g	Bond weakens 17% initially but ok over life cycle ?	NE-Lap Shear; T_g decreased 17%		25% stronger after fi exp but 20% weaker over life; T_g decrease 12%
EZE 240	Bond weakens 22% *X*		T_g decreased 25%		T_g decreased 12%
Intex 8125	Bond weakens 13% initially and same over life cycle		Bond weakens 12%; T_g decreased 16%	4.5% increase in volume; NE-T_g	Bond weakens 2.3%; 4% incr in wt; 2.5% i in volume; T_g decreas 60%
MSI 1025	Softens 5% immed but OK over life; 30% increase in strength of bond; NE-T_g.	Bond weakens 13%	T_g decreased 17%	1.3% increase in wt	
Oakite Liq Det #2	Bond weakens 30% *X*	Bond weakens 20% *X*	NE-Lap Shear; T_g decreased 27% *X*	1.3% increase in wt; NE-T_g	11% stronger bond
PF Degreaser	NE	Bond weakens 15% ?	T_g decreased 19%	NE-Lap Shear	T_g decreased 9%
Citranox			NE-Lap Shear		T_g decreased 7%
Water	Softens immed but OK over life; NE-T_g				Bond weakens 12%
1,1,1-Trichloroethane		Bond weakens 20% *X*	T_g decreased 40%	Softens immed; NE-Lap Shear, T_g *X*	10% stronger bond
Freon 113	NE-T_g		NE-Lap Shear; T_g decreased 17%	NE-T_g	Bond weakens 28%
General Comments about Adhesive Performance after Exposure	No sig change in wt or volume. Chem anal showed chem change and pickup of detergent. T_g increased.	No softening. T_g increased. Adhesive failure on lap shears. Weakest of the epoxies. No sig change in wt or vol. No chem changes.	No softening; no sig degradation in strength of bond; no appreciable dimensional changes; no chem changes.	Unexp sample softened over life cycle-others did not; wt decreases; no volume changes except 4; T_g increased; no chem changes	No softening; very strong bond except a noted; increase in volume with alkaline water; minor chemic changes-hydrolysis

Legend For Table 19

NE = Combination not evaluated.

NE-T_g = T_g analysis not performed on combination.

NE-Lap Shear = Lap shear analysis not performed on combination.

NE-Bulk = Bulk sample analysis not performed on combination.

***X* = Do not use! Degradation observed is statistically significant at 95% level.**

? = Do not use this combination without application-specific testing. Caution advised because of the degradation observed which

ble 19. **Summary of Results for Adhesive-Detergent Combinations for All Responses**

Epoxy					Sealant	
4/BA5	LCA4XM/BA5XM	C-7/Activator W	Epon 828/ Versamid 125	Eccobond 2216	Ablestik 724/14C	PR1422B
se in volume	2.6% increase in volume; 0.5% increase in wt; NE-T_g	T_g decreased 7%	T_g increased 24%	Softened immediately; T_g decreased 34%; 3% inc in wt X		
ar; T_g 7%		25% stronger after first exp but 20% weaker over life; T_g decreased 12% X	Softened immediately; T_g decreased 29%; bond weakens; 4% increase in wt X	16% stronger bond		
d 25%		T_g decreased 12%	Bond weakens 25% initially but OK over life; T_g decreased 21% ?	Softened immed, but OK over life; 10% stronger bond; NE-T_g		
ns 12%; T_g 6%	4.5% increase in volume; NE-T_g	Bond weakens 2.3%; 4% incr in wt; 2.5% incr in volume; T_g decreased 60% X	Bond weakens 22%; T_g decreased 21% X	Softened over life; bond weakens 14% X	10.8% increase in volume. X	4% incr in wt
d 17%	1.3% increase in wt		Bond weakens 15% initially but OK over life; T_g decreased 6% ?	20% stronger bond; NE-T_g		
ar; T_g 7% X	1.3% increase in wt; NE-T_g	11% stronger bond	Bond weakens 16%; T_g decreased 10%	17% stronger bond; NE-T_g	9.5% increase in volume. X	Softened immed.
d 19%	NE-Lap Shear	T_g decreased 9%		NE	9.8% increase in volume. X	
ar		T_g decreased 7%	Softened immed but OK over life; 4% increase in wt; T_g decreased 20% X	33% stronger bond; NE-T_g		
		Bond weakens 12%	Bond weakens 12%; T_g decreased 20% ?	Bond 10% weaker; NE-T_g		Softened.
d 40%	Softens immed; NE-Lap Shear, T_g X	10% stronger bond	Softened over life; 2% incr in wt X	Softened; 3% incr in wt X	9% increase in wt. X	
ar; T_g 7%	NE-T_g	Bond weakens 28% X	T_g decreased 17% ?	20% stronger bond; 4% decrease in wt	10% increase in volume X	Softened over life.
g; no sig in strength appreciable changes; no es.	Unexp sample softened over life cycle-others did not; wt decreases; no volume changes except 4; T_g increased; no chem changes	No softening; very strong bond except as noted; increase in volume with alkaline water; minor chemical changes-hydrolysis	Strongest bond except as noted; wt increases; volume decreases; minor chem changes-hydrolysis	Softer than other epoxies; maintained SH over life except as noted; strong bond except as noted; weight increases	No change in SH. Adhesive failure on lap shear. Decreases in weight. Significant distortion. No chem changes.	Unexp control harde over life; adhesive failure; softened and distorted considerabl decreased in wt; som chem changes

5% level.

Caution advised because of the degradation observed which was significant at the 80% level.

4.5.3 General Observations

The following conclusions were drawn from the results:

- Certain effects are present after life-cycle testing that do not appear following a single exposure. Thus, the method of repeated exposures is recommended for future adhesive evaluations.

- Little softening of the epoxies was noted (T_g). Table 18 summarizes the recommendations about epoxy-detergent combinations and decreased T_g.

- There is a need to model higher-order effects to improve predictive power. An in-depth study with one representative adhesive and one representative detergent, five levels of temperature, 3 frequencies of sonication, 3 durations of sonication, etc., to further understand the effect of cleaning on adhesives is recommended.

- Statistical analysis did not support the conventional wisdom that says there is a linear, main effect of temperature on all response measure. The only measure for which the effect was clear was strength of the bond. Bond strength decreased with increased temperature.

- 1,1,1-Trichloroethane and Freon 113 do cause changes in the exposed adhesives, and overall, their impact on the adhesive in a cleaning operation is comparable to aqueous cleaners.

- Aqueous and nonaqueous cleaning compounds perform similarly with respect to hardness, degradation in strength of bond, and other performance measures to solvent cleaners.

5.0 CONCLUSIONS

As discussed in Section 3.1, this study has used a screening design to allow the large number of independent variables selected to be run in the smallest possible matrix. This compaction of the independent variables results in a matrix in which many variables are changing simultaneously in each trial. Using statistical analysis, the independent variables can be analyzed for main effects and some information about interactions can be inferred. Please see Table 20. However, caution is advised against extension of results from a screening design beyond those cited in this report. These extensions are not statistically valid and can lead to erroneous results. The sections that follow have been written with these limitations in mind.

This section discusses main effects of the study which are valid over the range of parameters studied for all cases as well as presenting generalized conclusions. Section 6.0, *Recommendations*, presents tables listing favorable adhesive-detergent compositions (vs. unexposed adhesive controls) as related to the dependent/measured variables. These recommendations must be related on an individual basis to specific end use properties required for a given application in order to obtain the most benefit. The reader is reminded that trials that show significant degradation must be treated as single data points that cannot be directly related to other trials due to the confounding of the matrix independent variables.

Table 20. Summary of Data Analysis for Main Effects for Matrix

		Physical Dimension	Shore D Hardness	% Water	Lap Shear	Strength of Bond	T_g
Process	Temperature		No effects		No effects noted	High temperature lowered strength	No Effects or trends were observed
	Method	Sonication showed less change-possibly due to less exposure time	No effects		No effects noted	No effect	No Effects or trends were observed
	Life Cycle	Life cycle is important	No effects	Life cycle increased % water to a small extent	No effects noted	Life cycle negative effect interrelated with high temperature	Life cycle is important
Detergents			Detergents 3 and 8 caused increasing hardness over life cycle	PF Degreaser, 1,1,1-trichloroethane and Freon 113 all acted similarly showing weight gain	No effects noted	Detergent 4 causes decreased bond strength; detergents 9 and 10 show decreased strength versus unexposed controls; combinations of Adhesive 1 with Adhesive 6 produce lower bonds	No Effects or trends were observed
Adhesives		PR1422 and GE RTV60 are effected by exposure	Adhesive 7 shows softening over life cycle	Adhesive 8 showed significant % water gains and variation; Adhesives 9 and 10 increase weight slightly (5%) while Adhesive 4 shows a slight decrease in weight (1%)	No effects noted	Adhesive 2 loses strength after exposure; freon cleaning shows strength degradation over the life cycle for Adhesive 7 and to a lesser extent, with Adhesives 1, 2, and 9	No Effects or trends were observed

5.1 Extent of Cure Comment

The cure cycle selected for this study, 200 F for one hour, is not optimal for all of the adhesives studied. It does represent conditions in general use at AGMC and is therefore considered valid for this study. None of the adhesives showed poor lap shear values for unexposed control which indicates that any undercuring present did not show up in lap shear performance. Therefore, it was concluded that undercuring, if present, was not a factor in this study.

5.2 Sealant Observations

The sealant-type materials used in this study were GE RTV60, PR1422, and Ablestik 724/14C. These materials, in general, did not hold up as well as expected. PR1422 bulk exposures were stopped after 3 cycles as the degradation was too great to continue. GE RTV60 showed significant physical distortions toward the end of the life cycle testing. The samples did recover to their original dimensions in 24-48 hours after life cycle exposure. Ablestik 724/14C held up the best of the sealants. Lap shear values for these materials are low even under optimum conditions which use primers which were not used in this program. Therefore, analysis of the lap shear data must be viewed carefully as the materials were not being used under optimum conditions nor in a test normally used to judge their performance. Therefore, in the analysis, sealants were removed from consideration when it was deemed appropriate.

5.3 Overall Conclusions

The following list of overall conclusions and recommendations have been derived from the data analyses:

- The substances evaluated fall into two distinct categories, sealants and epoxies. In general, the sealants withstand cleaning less well than the epoxies.

- The elastomer sealant GE RTV60 does not withstand cleaning by any means. It loses dimensional stability and bonding strength upon exposure to any of the cleaning processes evaluated.

- The other sealants, Ablestik 724/14C and PR 1422B, do not retain bonding strength upon exposure to the cleaning solution-method-temperature combinations evaluated.

- The effect of exposure of epoxies to repeated cleanings is summarized in Table 21. The table presents indications of which combinations of detergent and adhesive are recommended and which are not recommended.

- In general, there was no significant difference in hardness level exhibited after repeated cleaning. Epoxies tended to maintain their hardness and sealants slightly increased in hardness.

- Preliminary analysis indicated that volumetric and weight changes observed fell well within the range of acceptability. Thus, the samples were exhibiting acceptable dimensional stability.

- Two adhesive families, fluid and low coefficient, have at least two members allowing evaluation of family traits. No generalized conclusions regarding cleaning-induced degradation were found in this study.

Table 21. Summary of Results for Adhesive-Detergent Combinations for All Responses

Detergent	Epoxy							Sealant		
	FA8/BA5	LCA9/BA5	LCA4/BA5	LCA4XM/BA5XM	C-7/Activator W	Epon 828/ Versamid 125	Eccobond 2216	Ablestik 724/14C	PR1422B	GE RTV60
Versaclean							Caution. Softening initially. Tg decreased.		Physical distortion.	
Brulin 815 GD				Bulk Effects. X	Caution: lap shear.	Caution: Softening water uptake (4%). Tg decreased lap shear.			Physical distortion.	
EZE 240	Caution: lap shear.								Physical distortion.	
Intex 8125				Caution: dimensional stability.	Caution: lap shear water uptake (4%). Tg decreased.	Caution: lap shear.	Caution: softens over lifetime.	Caution: dimensional stability +10.8% volume.	Physical distortion.	
MSI 1025				Bulk effects. X					Physical distortion.	
Oakite Liq Det #2	Caution: lap shear.	Caution: lap shear.						Caution: dimensional stability +9.8% volume.	Physical distortion.	
PF Degreaser	X	Caution: lap shear.		X				Caution: dimensional stability +9.8% volume.	Physical distortion.	Not recommended. Excessive softening. +13% water uptake.
Citranox				Bulk effects. X		Caution: softening initially. Water uptake (4%). Tg decreased.			Physical distortion.	
Water				Bulk effects. X	Caution: lap shear.				Physical distortion. Softening.	
1,1,1-Trichloro-ethane		Caution: lap shear. X		Caution: softens adhesive immediately.		Caution: softens over life cycle.	Caution: softening.	Caution: water uptake (9%)	Physical distortion.	Caution: softens immediately. Weight change +5%.
Freon 113					Caution: lap shear. Tg increases. X			Caution: dimensional stability +10% volume.	Physical distortion. Softening.	Not recommended. Weight change +28%.
General Comments about Adhesive Performance after Exposure	No significant changes in wt or dimensions. Chem analysis shows detergents pick-up and chem changes.	No chem changes. Lap shear shows degradation.	No chem changes. Very stable performance.		Increases in volumes with alkaline water exposure. Minor hydrolysis observed.	Weight increases. Volume decreases. Minor hydrolysis observed.		Significant bulk sample distortion. No chemical changes.	Extreme distortion observed. See text for discussion	Nonaqueous cleaners not recommended due to significant weight changes. Physical dimensional stability poor.

Table 21. Summary of Results for Adhesive-D

Detergent	Epoxy				
	FA8/BA5	LCA9/BA5	LCA4/BA5	LCA4XM/BA5XM	C-7/Activator W
Versaclean					
Brulin 815 GD				Bulk Effects. *X*	Caution: lap shear.
EZE 240	Caution: lap shear.				
Intex 8125				Caution: dimensional stability.	Caution: lap shear water uptake (4%). Tg decreased.
MSI 1025				Bulk effects. *X*	
Oakite Liq Det #2	Caution: lap shear.	Caution: lap shear.			
PF Degreaser	*X*	Caution: lap shear.		*X*	
Citranox				Bulk effects. *X*	
Water				Bulk effects. *X*	Caution: lap shear.
1,1,1-Trichloro-ethane		Caution: lap shear. *X*		Caution: softens adhesive immediately.	
Freon 113					Caution: lap shear. Tg increases. *X*
General Comments about Adhesive Performance after Exposure	No significant changes in wt or dimensions. Chem analysis shows detergents pick-up and chem changes.	No chem changes. Lap shear shows degradation.	No chem changes. Very stable performance.		Increases in volumes with alkaline water exposure. Minor hydrolysis observed.

Table 21. Summary of Results for Adhesive-Detergent Combinations for All Responses

	Epoxy					Sealant		
	LCA4/BA5	LCA4XM/BA5XM	C-7/Activator W	Epon 828/ Versamid 125	Eccobond 2216	Ablestik 724/14C	PR1422B	
					Caution. Softening initially. Tg decreased.		Physical distortion.	
		Bulk Effects. X	Caution: lap shear.	Caution: Softening water uptake (4%). Tg decreased lap shear.			Physical distortion.	
							Physical distortion.	
		Caution: dimensional stability.	Caution: lap shear water uptake (4%). Tg decreased.	Caution: lap shear.	Caution: softens over lifetime.	Caution: dimensional stability +10.8% volume.	Physical distortion.	
		Bulk effects. X					Physical distortion.	
						Caution: dimensional stability +9.8% volume.	Physical distortion.	
		X				Caution: dimensional stability +9.8% volume.	Physical distortion.	N E -
		Bulk effects. X		Caution: softening initially. Water uptake (4%). Tg decreased.			Physical distortion.	
		Bulk effects. X	Caution: lap shear.				Physical distortion. Softening.	
X		Caution: softens adhesive immediately.		Caution: softens over life cycle.	Caution: softening.	Caution: water uptake (9%)	Physical distortion.	C i V
			Caution: lap shear. Tg increases. X			Caution: dimensional stability +10% volume.	Physical distortion. Softening.	N V
.	No chem changes. Very stable performance.		Increases in volumes with alkaline water exposure. Minor hydrolysis observed.	Weight increases. Volume decreases. Minor hydrolysis observed.		Significant bulk sample distortion. No chemical changes.	Extreme distortion observed. See text for discussion	N r si P st

ve-Detergent Combinations for All Responses

			Sealant		
W	Epon 828/ Versamid 125	Eccobond 2216	Ablestik 724/14C	PR1422B	GE RTV60
		Caution. Softening initially. Tg decreased.		Physical distortion.	
ear.	Caution: Softening water uptake (4%). Tg decreased lap shear.			Physical distortion.	
				Physical distortion.	
ear %).	Caution: lap shear.	Caution: softens over lifetime.	Caution: dimensional stability +10.8% volume.	Physical distortion.	
				Physical distortion.	
			Caution: dimensional stability +9.8% volume.	Physical distortion.	
			Caution: dimensional stability +9.8% volume.	Physical distortion.	Not recommended. Excessive softening. +13% water uptake.
	Caution: softening initially. Water uptake (4%). Tg decreased.			Physical distortion.	
ar.				Physical distortion. Softening.	
	Caution: softens over life cycle.	Caution: softening.	Caution: water uptake (9%)	Physical distortion.	Caution: softens immediately. Weight change +5%.
ar. *X*			Caution: dimensional stability +10% volume.	Physical distortion. Softening.	Not recommended. Weight change +28%.
nes er ed.	Weight increases. Volume decreases. Minor hydrolysis observed.		Significant bulk sample distortion. No chemical changes.	Extreme distortion observed. See text for discussion	Nonaqueous cleaners not recommended due to significant weight changes. Physical dimensional stability poor.

6.0 RECOMMENDATIONS

The recommendations for this study are made for adhesive-detergent combinations and are presented in Table 21. In most cases, overall trends were not evident and degradation when observed was associated with very specific aspects of adhesive performance. Therefore, the performance characteristics which are critical for a given end-use application must be identified and then compared to the recommendation chart to determine if a potential problem exists. The matrix used in this study is a screening design and therefore not all combinations were evaluated. This is noted in the table accordingly. No entry indicates that degradation is not expected with this combination.

The process conditions to avoid are temperatures above 135 F and cumulative exposure times exceeding 12 hours over the service life of the item to be cleaned. Of the adhesives studied, the most stable is LCA4/BA5 which works well with all of the detergents studied. The sealant samples, Ablestik 724/14C, PR1422B, and GE RTV60 perform poorly with regards to weight and physical dimension changes, and cleaning of these systems over a 12 exposure life cycle is not recommended.

Of the detergents studied, Intex 8125 should be used with caution. The nonaqueous detergents, 1,1,1-trichloroethane, Freon 113 and PF Degreaser were associated with softening, increases in weight, and decreased lap shear and in selected cases caused more degradation than many of the aqueous cleaners. EZE 240 and Versaclean are the detergents recommended for most cleaning needs because they do not affect any adhesive characteristic which is of practical concern.

Lap shear and percent weight change were the most critical and significant adhesive factors found in this program and should form the basis of any future work. PA FT-IR, which was shown to be an excellent tool for determining chemical changes in adhesive chemistry, and a smaller, more focused matrix studying immersion exposures at moderate temperatures over a simulated life cycle, are recommended for future activities.

7.0 REFERENCES

Box, George E.P., William G. Hunter, and J. Stuart Hunter, *Statistics for Experimenters: An Introduction to Design, Data Analysis, and Model Building*. New York: John Wiley & Sons, 1978.

Lee, Henry, and Kris Neville, *Handbook of Epoxy Resins*. McGraw Hill, Inc., 1967.

Cornstock, M. Joan, Editor, *Epoxy Resin Chemistry II*. American Chemical Society, 1983.

Brinson, Hal, Editor, *Engineered Materials Handbook, Adhesives and Sealants*, Volume 3. American Society of Materials International, 1990.

7.1 ASTM Test Methods

ASTM D1002	Standard Test Method for Strength Properties in Shear by Tension Loading
ASTM D1151-90	Effect of Moisture and Temperature on Adhesive Bond
ASTM D896-90	Standard Test Method for Resistance of Adhesive Bonds to Chemical Reagents
ASTM D1183-70	Standard Test Methods for Resistance of Adhesive to Cyclic Laboratory Aging Conditions
ASTM D570-81	Water Absorption of Plastics

APPENDIX A

DESIGN MATRIX IN TRIAL ORDER

Appendix A
Design Matrix in Trial Order

The following tables in this appendix describe the levels and types of independent variables used for each trial. At the bottom of the tables is a legend describing the variable codes. As an example, Trial 1 uses Adhesive 5 (Ablestik 724/14C), C3 (Freon 113) detergent, and UL (25 kHz) ultrasonic bath cleaning conducted at room temperature.

Trial	X1=Adhesive	X2=Detergen	X3=Method	X4=Temp	X5=Block
1	5	C3	UL	1	1
2	9	5	UL	3	1
3	0	8	UH	1	1
4	8	7	S	1	1
5	2	6	UL	2	1
6	6	2	UH	3	1
7	0	5	S	1	1
8	1	1	UL	3	1
9	8	2	S	2	1
10	6	C3	S	1	1
11	4	2	UH	1	1
12	9	7	S	2	1
13	9	C4A	UH	2	1
14	7	C1	UH	2	1
15	0	C4B	UH	1	1
16	1	C4A	S	3	1
17	2	3	S	3	1
18	8	5	UL	2	1
19	9	2	S	3	1
20	7	4	S	3	1

C1 = distilled water
C2 = 1,1,1-trichloroethane
C3 = Freon 113
C4A = acidified distilled water
C4B = alkaline-adjusted distilled water

UL = sonication at 25 kHz
UH = sonication at 67 kHz
S = soaking
X4 (1) = room temperature
X4 (2) = 135 F
X4 (3) = 190 F

Trial	X1=Adhesive	X2=Detergen	X3=Method	X4=Temp	X5=Block
21	5	C2	S	1	1
22	8	C4B	UH	1	1
23	6	7	UL	2	1
24	6	6	S	3	1
25	4	C2	S	1	1
26	3	6	UL	3	1
27	2	C2	UH	1	1
28	5	2	UH	2	1
29	9	3	UL	1	1
30	2	8	S	2	1
31	8	C3	S	1	1
32	1	3	S	2	1
33	3	8	S	3	1
34	4	4	UH	3	1
35	2	4	UH	1	1
36	6	4	S	2	1
37	7	5	UL	1	1
38	7	2	S	1	1
39	0	3	UL	2	1
40	3	4	UH	2	1

C1 = distilled water
C2 = 1,1,1-trichloroethane
C3 = Freon 113
C4A = acidified distilled water
C4B = alkaline-adjusted distilled water

UL = sonication at 25 kHz
UH = sonication at 67 kHz
S = soaking
X4 (1) = room temperature
X4 (2) = 135 F
X4 (3) = 190 F

Trial	X1=Adhesive	X2=Detergen	X3=Method	X4=Temp	X5=Block
41	8	C1	UH	3	1
42	6	C4B	S	2	1
43	4	1	S	3	1
44	1	C2	UH	1	1
45	3	C2	S	1	1
46	3	1	S	2	1
47	5	4	S	1	1
48	7	7	UL	3	1
49	4	C3	UL	1	1
50	1	6	UL	1	1
51	2	1	S	1	1
52	9	C1	S	1	1
53	0	7	S	3	1
54	5	7	UL	1	1
55	5	6	S	2	1
56	4	6	S	1	1
57	6	C1	UH	1	1
58	1	8	S	1	1
59	3	C3	UL	1	1
60	0	C1	S	2	1

C1 = distilled water
C2 = 1,1,1-trichloroethane
C3 = Freon 113
C4A = acidified distilled water
C4B = alkaline-adjusted distilled water

UL = sonication at 25 kHz
UH = sonication at 67 kHz
S = soaking
X4 (1) = room temperature
X4 (2) = 135 F
X4 (3) = 190 F

Trial	X1=Adhesive	X2=Detergen	X3=Method	X4=Temp	X5=Block
61	9	8	S	3	2
62	7	C4B	S	3	2
63	5	C4A	S	1	2
64	0	6	S	1	2
65	8	8	S	2	2
66	2	5	UH	2	2
67	0	1	S	3	2
68	9	1	S	2	2
69	9	4	UL	2	2
70	1	5	UH	1	2
71	8	3	S	3	2
72	5	1	UH	1	2
73	0	2	UL	1	2
74	4	C1	S	2	2
75	3	C1	S	1	2
76	0	C3	UH	1	2
77	5	8	UL	2	2
78	5	5	S	2	2
79	5	3	UH	3	2
80	9	6	UH	3	2

C1 = distilled water
C2 = 1,1,1-trichloroethane
C3 = Freon 113
C4A = acidified distilled water
C4B = alkaline-adjusted distilled water

UL = sonication at 25 kHz
UH = sonication at 67 kHz
S = soaking
X4 (1) = room temperature
X4 (2) = 135 F
X4 (3) = 190 F

Trial	X1=Adhesive	X2=Detergen	X3=Method	X4=Temp	X5=Block
81	2	C1	UL	3	2
82	7	6	UH	1	2
83	5	C1	S	3	2
84	2	7	S	1	2
85	7	C2	UL	1	2
86	2	2	S	2	2
87	6	C2	UL	1	2
88	7	1	UH	3	2
89	6	3	S	1	2
90	3	3	UH	1	2
91	1	C3	S	1	2
92	1	7	UH	3	2
93	8	C2	UL	1	2
94	9	C2	S	1	2
95	3	C4B	UL	2	2
96	4	8	UL	1	2
97	8	1	S	1	2
98	4	3	UH	2	2
99	3	5	UH	3	2
100	4	C4A	UL	3	2

C1 = distilled water
C2 = 1,1,1-trichloroethane
C3 = Freon 113
C4A = acidified distilled water
C4B = alkaline-adjusted distilled water

UL = sonication at 25 kHz
UH = sonication at 67 kHz
S = soaking
X4 (1) = room temperature
X4 (2) = 135 F
X4 (3) = 190 F

Trial	X1=Adhesive	X2=Detergen	X3=Method	X4=Temp	X5=Block
101	2	C3	S	1	2
102	6	5	S	3	2
103	6	1	UH	2	2
104	3	7	S	2	2
105	4	5	S	1	2
106	4	7	S	3	2
107	1	C1	UL	2	2
108	6	8	UL	3	2
109	0	4	UL	3	2
110	7	C3	S	1	2
111	2	C4B	UL	1	2
112	0	C2	S	1	2
113	8	6	UH	2	2
114	8	4	UL	1	2
115	1	2	S	1	2
116	3	2	S	3	2
117	9	C3	UH	1	2
118	7	8	S	1	2
119	7	3	S	2	2
120	1	4	S	3	2

C1 = distilled water
C2 = 1,1,1-trichloroethane
C3 = Freon 113
C4A = acidified distilled water
C4B = alkaline-adjusted distilled water

UL = sonication at 25 kHz
UH = sonication at 67 kHz
S = soaking
X4 (1) = room temperature
X4 (2) = 135 F
X4 (3) = 190 F

APPENDIX B

MATRIX UNIT OPERATION PROCEDURES

Appendix B
Matrix Unit Operation Procedures

The following procedure and operations were used to conduct individual activities required to implement the matrix. The references are to sections of the test plan prepared for this program.

1) Refer to matrix worksheet and select correct adhesive. Enter all data on trial worksheet.

2) Conduct procedure per Test Schedule and operational Block Schedule

3) Prepare test samples per 5.2.2. Prepare 12 lap shears and cast 4 1"x3" bulk samples. Six of the lap shears and 2 bulk samples will be used as unexposed controls and will be stored in a controlled environment per 5.2.3c.

4) Cure samples per 5.2.3.

5) Refer to matrix worksheet for exposure per 5.2.4.

6) Dry samples per 5.2.5. Conduct 5.2.7 and 5.2.8 on both exposed and unexposed samples.

7) Complete Life Cycle Testing per 5.2.6. If early termination is required, record observations and termination cycle on trial worksheet.

8) Conduct 5.2.7 and 5.2.8 on Life Cycle and unexposed samples.

5.2.4 <u>Exposure to Cleaning Agents</u>

a. Clearly mark all samples by inscribing the trial and replicate number on the film or the lap shear plate. If this is impractical, the sample will be placed in a mesh bag and a separate labeling plate containing this information shall be attached.

b. The cleaning solution shall be prepared one hour prior to the exposure per the manufacturer's specification. Record pH at room temperature and then heat solution to the temperature specified in the matrix worksheet in Appendix A.

c. Where possible, all of the samples in a given block should be exposed at the same time.

d. The exposure process designated in the matrix worksheet shall be conducted. The exposure process will consist of either immersion for one hour or sonication for five minutes. Record the temperature to $\pm$ 2 F before and after the cleaning procedure. This will be followed by five minutes immersion in room temperature clean deionized water as a rinse.

e. Initial drying will consist of patting the samples dry and then placing them in a 130 F forced air convection oven for thirty minutes. Any pre-drying testing shall be done following this procedure. If no testing is to be conducted at this point, this step will not be conducted.

f. At the end of the cycle, observe the cleaning tank for any residue or appearance changes and record observations.

5.2.5 Drying Cycle

a. After exposure, the samples will be dried for one hour at 160 F at 60 $\pm$ 20 mm Hg vacuum.

b. The dried samples will be allowed to equilibrate 16 to 24 hours in a constant temperature room prior to further operations. See 5.2.3c.

5.2.6 Life Cycle Testing

a. Life cycle testing shall consist of twelve total cycles of cleaning agent exposure, drying, and dwell time between exposure.

b. Within a given trial, the cleaning operation will not be varied during the life cycle.

c. The life cycle testing will be terminated at any time if the samples have visually degraded to the extent that evaluation of the samples would not be possible if further cycling were conducted. The samples will evaluated as though they had completed the entire twelve cycles and the number of cycles completed will be recorded. The initial method for consideration of life cycle test termination will be visual inspection. The conditions required to terminate cycling shall be loss or gain of more than 10% by weight of the bulk samples and any separation observed in the lap shears. Other conditions may also justify termination and will be recorded.

5.2.7 Bulk Effect Testing Methods

a. Duplicate bulk samples will be used.

b. Dimensional stability will be evaluated per ASTM D543 at intervals as described in the Testing Schedule.

c. Hardness will be determined using a Shore A or D test gauge applied manually to the bulk samples at intervals as described in the Testing Schedule and ASTM D2240. Film samples will be placed on a flat piece of chrome steel with a minimum thickness of 0.25" for measurement. A Shore measurement stand may be substituted.

d. Water absorption will be determined per ASTM D 570 at intervals in accordance with (IAW) the Testing Schedule. Weighing will be conducted to three digit accuracy.

e. Samples for thermal analysis will be taken from the end of one of the bulk samples as needed. The same sample will be used as a source for all thermal sampling needs. The area to be sampled will be chosen to be representative of the average condition of the film. The apparent T_g and the extent of cure will be determined and recorded IAW the Testing Schedule. Differential Scanning Calorimetry (DSC) data will be obtained on one sample per trial from room temperature to 400 F. A Perkin Elmer System 7 and its associated software will be used to collect and analyze the data.

f. The appearance of the bulk sample will be evaluated visually for changes in color, surface roughness, flaking, crazing and other irregularities IAW the Testing Schedule. The results will be recorded. Photographic documentation of degradation observed in the bulk samples will be made.

5.2.8 Interfacial Testing

a. Three replicate samples will be prepared for each measurement of interfacial properties required in the Testing Schedule. This testing will be conducted IAW ASTM D1002 and the values recorded.

b. The bondline will be controlled at 5 mils by placing 5 mil glass beads in the wet bondline prior to assembly of the bond. The bond will be clamped with spring clamps during the assembly and curing of the samples per the current AGMC technique. Excess adhesive squeezed out during assembly will be physically removed prior to curing.

c. The type of bond failure as well as the strength of the bond will be recorded. Failure will be classified using the following criteria.

Adhesive - Adhesive is pulled cleanly from the bonded area with no residue over > 90% of the area observed

Cohesive - Adhesive remains on both test coupons with failure occurring in the adhesive interlayer over > 90% of the area observed

Delamination - A partial failure having areas of both adhesive and cohesive failure, a rating of the amount of cohesive failure present from 10-90% is assigned

d. Aluminum 2024 plate stock chemically etched will be used to assemble the lap shears. The chemical etching technique will be performed as described by Bacon Industries (see Appendix B) and coupons so treated will be used within 72 hours.

5.2.9 Chemical Analysis

a. A series of follow on activities will be conducted with two adhesive and cleaning agent combinations exhibiting degradation and two combinations which are not showing degradation. These combinations will be selected from the first block's trials. These activities will be conducted concurrently with the implementation of the second block's experiments.

b. IR analysis using photoacoustic FTIR will be performed on degraded/non-degraded samples as is after life time cycling has been completed. Photoacoustic FTIR will be applied to the solid samples without further processing. Both bulk and interfacial samples will be evaluated as appropriate. Areas displaying appearance changes in particular will be examined. Specifically, the results of this analysis will be used to document and identify residual cleaning agents in the adhesive matrix, changes in the cured adhesives chemistry, and formation of new functional groups in the adhesive layer.

c. Thermal analysis will be performed on the same samples selected for B above. Thermal Mechanical Analysis and additional DSC testing will be conducted to quantify and determine the extent of property degradation of these samples.

AGMC Aluminum Etching Procedure

1. Prepare the bath as follows:

4461-1620	Distilled Water	2740 ml
46361	Concentrated H_2SO_4	560 ml
	$Na_2Cr_2O_7$	300

Exercise extreme caution: highly corrosive

2. Obtain clean acetone. Carefully soak plates in acetone for 5 minutes. Remove and allow to dry in air for 15 mins.

3. Heat bath solution (1) to 150 F ± 10 F.

4. Suspend aluminum plates in solution so they do not touch each other and that at least 1 inch and not more than 2 inches of the plate is covered by the etching bath.

5. Expose aluminum plate samples for 12 minutes.

6. Remove plates immediately into a large beakers filled with tap water. Running water (warm) will be used as a flush rinse for 15 minutes.

7. Immediately soak plates in clean distilled water for 2 minutes (minimum).

8. Allow to air dry on clean, lint-free towels.

APPENDIX C

DETAILED ANALYSIS RESULTS

Appendix C
Detailed Analysis of Results

This appendix presents some of the results of the analyses at a detailed level. These results complement and support the results and conclusions presented in the main body of the report. Presentation of these results is organized as follows:

- Section C.1 Analysis of Lap Shear Data
- Section C.2 Analysis of Bulk Sample Data
- Section C.3 Chemical Analysis

C.1 Analysis of Lap Shear Data

Results discussed in Section 4.2 were obtained by multiple regression analysis. The regression equation of L-PSI on indicators of the design variables compared against a control for each variable is shown below.

$$
\begin{aligned}
\text{L-PSI} = {} & 2726 + 55.8\,A_1 - 326.0\,A_2 + 19.6\,A_4 - 1788\,A_5 - 2612\,A_6 + \\
& 849.0\,A_7 + 750.0\,A_9 + 299.0\,A_{10} + 71.4\,D_1 - 140.0\,D_2 + \\
& 13.0\,D_3 - 122.0\,D_4 + 71.0\,D_5 - 102.0\,D_6 + 5.0\,D_7 + \\
& 116.0\,D_8 - 208.0\,D_9 - 195.0\,D_{10} - 130.0\,D_{11} + 96.0\,D_{12} + \\
& 17.0\,D_{13} - 25.7\,M_{UL} + 76.2\,M_{UH} - 124.0\,T_2 - 151.0\,T_3 - \\
& 316.0\,G_1 + 174.0\,G_2 - 46.1\,G_3 - 3.6\,G_4 + 1.0\,G_5
\end{aligned}
$$

where A_i = 1 when the adhesive is i
= 0 otherwise
D_j = 1 when the detergent is j
= 0 otherwise
M_k = 1 when the cleaning method is k
= 0 otherwise
T_l = 1 when the temperature is at level l
= 0 otherwise
G_m = 1 when the group is m
= 0 otherwise

Selected controls were Adhesive 3 (LCA4/BA5), no detergent (unexposed), soaking as the cleaning method, room temperature, and Group 6. The group effect described in Section 4.2 was observed in this analysis. As was obvious from the table of means, Adhesives 5 (Ablestik 724/14C), 6 (PR1422B), and 8 (GE RTV60) [the sealants] had far less strength than the other adhesives. Adhesive 2 (LCA9/BA5) had significantly less strength than the control, LCA4/BA5. Adhesives 7 (Adhesive C-7/Activator W), 9 (Epon 828/Versamid 125), and 10 (Eccobond 2216) had significantly greater strengths than Adhesive 3 (LCA4/BA5). The main effects of adhesive on L-PSI when compared to LCA4/BA5 are shown in Figure C-1. The result for Adhesive 7 (Adhesive C-7/Activator W) is most highly significant (t=8.70), Adhesive 9 (Epon 828/Versamid 125) is also quite significant (t=6.83), and Adhesive 10 (Eccobond 2216) is solidly significant (t=2.88).

This is a comparison of force required to break the lap shear samples for each adhesive using LCA4/BA5 (Adhesive 3) as the baseline. Adhesive 2 is the weakest epoxy, while 7, 9, and 10 are the strongest. The sealants, 5 and 6, have very poor bonding strength. The baseline (0 change) represents 2726 psi.

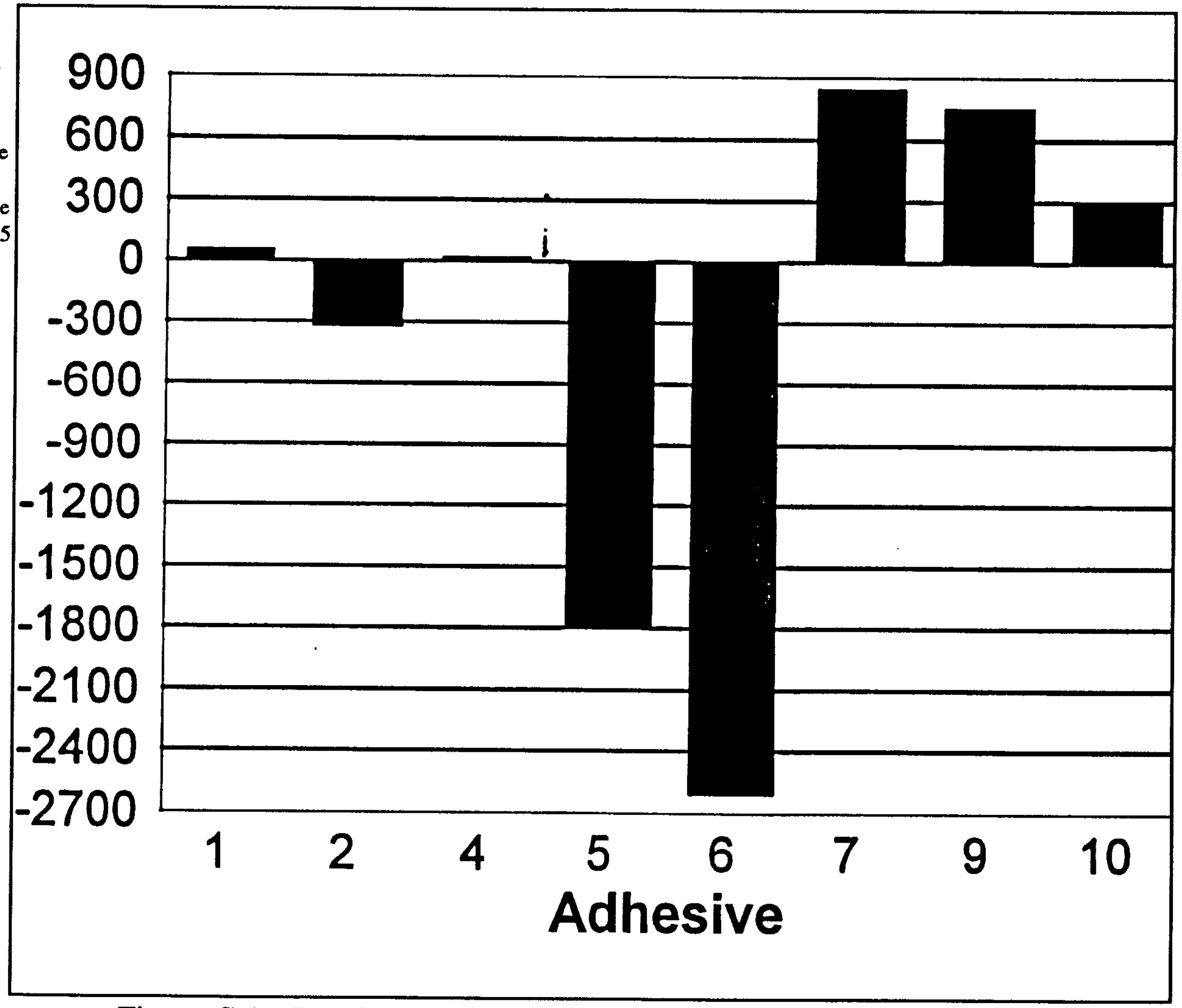


Figure C-1. Lap Shear Main Effects of Adhesive on Strength of Bond

"t" is a statistic used to infer whether an observed difference is indeed a difference in the response (presumably due to the factors investigated in the experiment) or simply a manifestation of the random variation in the response due to factors outside the design. For the number of trials in this analysis, $|t| \geq 1.96$ indicates statistical significance at the 95% level. When $|t| = 1.96$, the observed difference could be expected to occur by chance less than 5% of the time. A larger $|t|$ value indicates even less of a chance that the difference occurred at random and provides increased confidence in the conclusion that a true effect has been observed.

C.1.1 Analysis of Epoxies

Adhesives 5 (Ablestik 724/14C), 6 (PR1422B), and 8 (GE RTV60)—the sealants—have such low lap shear values that further meaningful analysis based on this response is not warranted. For this reason, the sealants were eliminated from the data set and the analysis of main effects was repeated. The analysis also showed that Adhesive 2 (LCA9/BA5) performed significantly worse and Adhesives 7 (Adhesive C-7/Activator W), 9 (Epon 828/Versamid 125), and 10 (Eccobond 2216) performed significantly better than the control. Some detergent effects were noted, and these are shown in Figure C-2. The histogram shows how strength of bond varies with each detergent in comparison to the baseline value of 2726 psi. While the differences are seen, only a change in excess of 190 psi (about 7%) is statistically significant. Thus, the two solutions that showed poor performance were water and 1,1,1-trichloroethane. The regression accounted for approximately 60 percent of the variability in the data. There was no effect of method or temperature in this analysis.

The life-cycle results showed similar trends. Detergent 4 (Intex 8125) still performed less well than the others but the difference fell just below the significance level. No other detergent effects were significant, including those noted for single exposure results. Although method remained nonsignificant, the effect of temperature appeared significant with increase in temperature being associated with decreasing bonding strength.

Change in force required to break lap shears for each detergent using the no-exposure condition as the control. The baseline (0 change) represents 2726 psi.

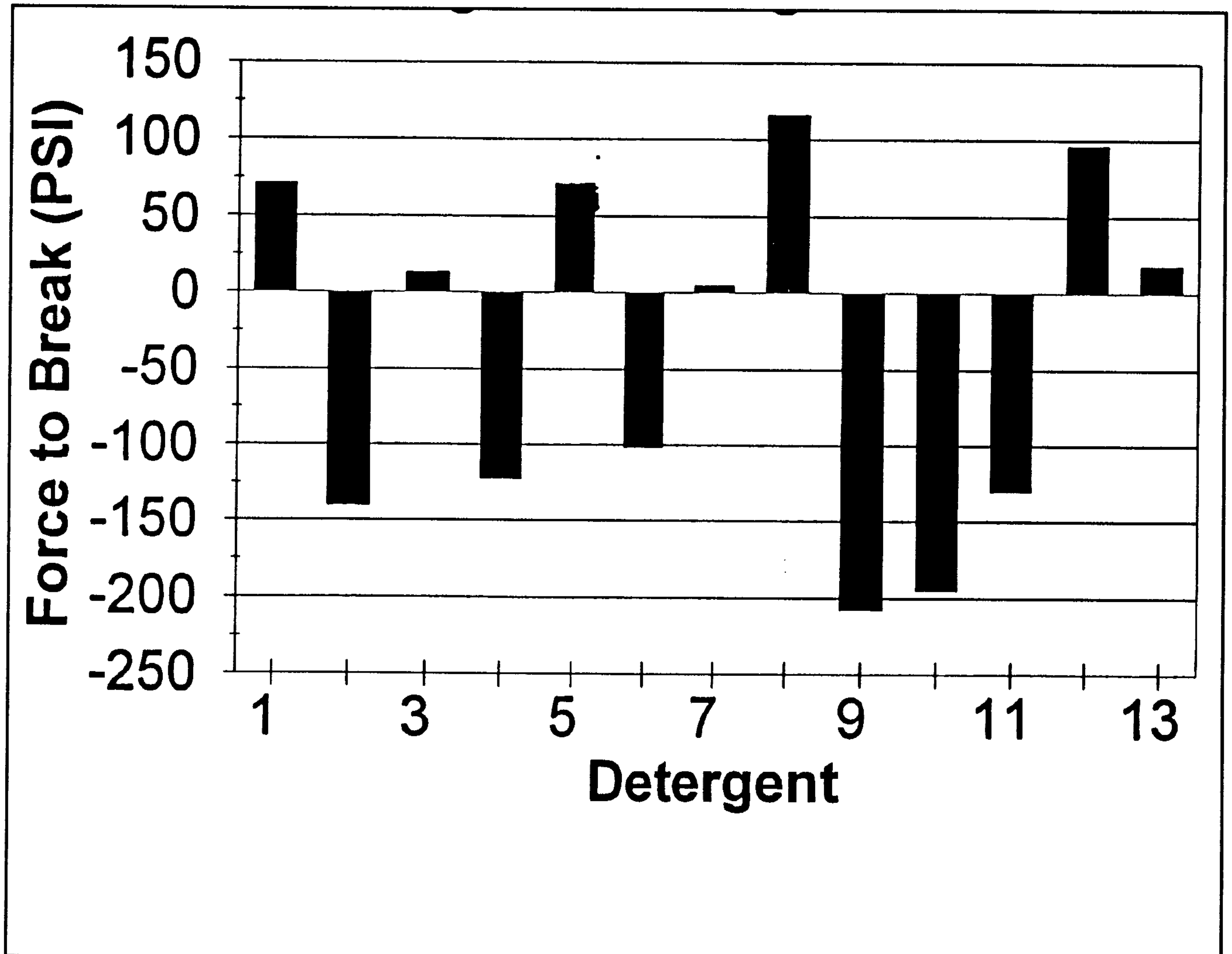


Figure C-2. Lap Shear Main Effects of Detergent on Strength of Bond

C.1.2 Analysis of LCA4 Full Exposure Matrix

The design was modified between Blocks I and II to allow all combinations of LCA4/BA5 and Versaclean with all cleaning methods and temperatures. The analysis on the LCA4/BA5 data progresses through detergents, method, frequency of sonication, and temperature. The detergent was eliminated as a variable and the focus was on method and temperature. No significant results were discovered, because there was essentially no degradation in bond strength for this adhesive-detergent combination for the methods and temperature range selected.

C.1.3 Analysis of Interaction Effects

An analysis was performed to investigate the detergent-adhesive effects, independent of method or temperature. A series of analyses was performed and the significant or near-significant results are presented in Table C-1. The numbers in the cells within the table are the t values for the coefficients fitted to the adhesive x detergent term in the equation. A t value greater than 1.96 or less than -1.96 is significant at the 95 percent level for these data. $|t|$ greater than 1.65 indicates significance at the 90 percent level (or 95 percent in a 1-tailed test). $|t|$ greater than 1.282 indicates significance at the 80 percent level (90 percent for 1-tailed test). That being the case, values without parentheses indicate statistical significance (at the 95 percent level). Values within parentheses indicate significance at the 90 percent level and values in [ ] indicate significance only at the 80 percent level. All comparisons are made to unexposed control samples.

Table C-1. Adhesive-Detergent Interactions (Lap Shear)

Detergent	Adhesive 1	2	3	4	7	9	10
Single Exposure							
Versaclean	-2.03			(-1.72)	2.47		[1.49]
Brulin 815 GD	1.99	-2.06	NE		[1.60]		
EZE 240					2.34	-2.96	
Intex 8125	[1.64]	(1.84)	(1.88)	2.41	-4.31	[-1.34]	[1.34]
MSI 1025	2.40					-2.70	
Oakite Liq. Det. #2	-3.40		NE	(-1.72)	4.23		3.20
PF Degreaser	NE			NE		[-1.38]	[1.45]
Citranox			NE		1.63		(1.84)
Water	3.16					-2.46	
1,1,1-Trichloroethane				NE	[1.61]	[1.36]	
Freon 113			NE				
Life-Cycle							
Versaclean							[1.26]
Brulin 815 GD			NE	[1.35]	-2.14	-3.16	2.19
EZE 240	[-1.63]						2.04
Intex 8125	2.57	2.37	[1.23]	2.11	-2.70	-2.60	
MSI 1025	2.15	[-1.23]					2.50
Oakite Liq. Det. #2	-2.58	(-1.67)	NE		2.76		3.75
PF Degreaser	NE	-2.09		NE	NE	[1.40]	NE
Citranox			NE			[-1.25]	2.42
Water			2.11		[-1.58]	[-1.60]	(1.89)
1,1,1-Trichloroethane				NE	1.94		
Freon 113	(1.72)	(1.71)	NE		-3.74	[-1.27]	2.45

NE	= combination was not evaluated
n.nn	= significant at 95% level (| t | ≥1.96)
(n.nn)	= significant at 90% level (1.96> | t | ≥1.65)
[m.mm]	= significant at 80% level (1.65> | t | ≥1.28)
blank cell	= no significant difference from unexposed control

One interesting result is that the combination of FA8/BA5 and Brulin 815GD seems to be especially good for a single exposure (significantly better than the control), while there is no difference over the simulated life cycle as is shown in Table C-2. The combination of FA8/BA5 and Intex 8125 is almost significantly worse than others after one exposure but it is significantly better after the simulated life cycle. The combination of FA8/BA5 and MSI1025 seems quite acceptable throughout, whereas FA8/BA5 with Oakite Liquid Detergent #2 is significantly worse than the controls. The combination of Eccobond 2216 and Oakite Liquid Detergent #2 appears particularly good for both single exposure and 12 exposures. Likewise, Citranox seems good in combination with Eccobond 2216. It is also good with C-7/Activator W for single exposure, but the difference subsides over the life-cycle.

Table C-2. Life-Cycle Recommendations About Adhesive-Detergent Combinations for Lap Shear (vs. Unexposed Control)

Detergent	Adhesive						
	1	2	3	4	7	9	10
Versaclean	caution			caution			
Brulin 815 GC		caution	NE		worst	worst	best
EZE 240	poor						best
Intex 8125	best	best		best	worst	worst	
MSI 1025	best					caution	best
Oakite Liq. Det. #2	worst	poor	NE	caution	best		best
PF Degreaser	NE	worst		NE	NE		NE
Citranox			NE				best
Water						caution	good
1,1,1-Trichloroethane				NE	good		
Freon 113	good	good	NE		worst		best

No entry = bonding strength not degraded significantly over life cycle
best = significantly stronger than unexposed controls over life cycle
good = somewhat stronger than unexposed controls over life cycle
poor = somewhat less strong than unexposed controls over life cycle
worst = significantly less strong than unexposed controls over life cycle
caution = significantly less strong after a single exposure but no significant difference over life cycle.

Only in one case—that is in combination with C-7/Activator W over the life cycle—is 1,1,1-trichloroethane better than other cleaning solutions. On the other hand, Freon 113 is associated with a significant degradation in strength in combination with C-7/Activator W over the life cycle. From this, it is concluded that the commonly used nonaqueous cleaners are not necessarily better than alternatives, and some are worse.

C.2 Analysis of Bulk Sample Data

This section provides detailed analysis of the following bulk sample responses:

- **Percentage Water Absorption** measured as change in weight (dWt) after a single exposure (SdWt) or over the life cycle (LdWt)
- **Dimensional Changes** measured as change in volume (dV) following a single exposure (SdV) or the simulated life cycle (LdV)
- **Hardness** measured as Shore A or Shore D (SH) following a single response (SSH) or the simulated life cycle (LSH).

Refer to Section 4.3 in the main body of the text for other results related to these responses.

C.2.1 Percentage Water Absorption

Adhesive 8 (GE RTV60) showed considerable variability (-1 to 33 percent) and weight gain far in excess of the control (Adhesive 3, LCA4/BA5). This change occurred with the initial exposure, the range being -0.38% to 29.0%. Further change over the life cycle was minimal. Adhesive 8 (GE RTV60) should not be cleaned with any aqueous based solutions.

Adhesive 6 (PR1422B) showed a decrease in weight after initial exposure (SdWt) and a much larger decrease over the life cycle (LdWt). However, this change was not a function of the exposure to cleaning because the unexposed control samples behaved

similarly. Therefore, weight change is not a reason to rule out aqueous cleaning of Adhesive 6 (PR1422B).

Analysis of the change in weight after 12 exposures resulted in the following observations. Adhesives 9 (Epon 828) and 10 (Eccobond 2216) tended to increase in weight more than the others, while Adhesive 4 (LCA4XM/BA5XM) showed a statistically significant tendency to decrease in weight. The range of this effect is very small, with the increases being less than 5 percent and the decreases of the order of 1-3 percent.

The effect of Detergent 7 (PF Degreaser) on increasing weight was significant when compared to the unexposed condition. Similarly, nonaqueous controls, 1,1,1,-trichloroethane and Freon 113, were significant in their association with increased weight. One could conclude that these three nonaqueous solutions had a similar effect and one that was distinctly different from the other solutions, all of which were aqueous.

C.2.1.1 Interaction Effects

An analysis of the adhesive-detergent interactions revealed that there was essentially no change in weight for Adhesives 1-3 (FA8/BA5, LCA9/BA5, and LCA4/BA5). LCA4XM/BA5XM increased 1.3% in weight with MSI 1025 and with Oakite Liquid Detergent #2 and 2.6% with Versaclean. PR1422B actually loses weight, indicating the presence of solvents that continue to evaporate over the life cycle. Washing accelerates this process slightly.

The other epoxies gained weight only in a few cases, compared to 0.5% gain for unexposed control:

- C-7/Activator W
  - Intex 8125 — 4.3%
  - Alkaline-adjusted distilled water — 3.8%

- Epon 828/Versamid 125
  - Brulin 815 GD — 4.3%
  - Citranox — 4.3%

Eccobond 2216 decreased in weight by 4% when exposed to Freon 113 and increased 3% after exposure to Versaclean, compared to 0.4% for the unexposed control. No other weight changes were noted.

Ablestik 724/14C absorbed 1,1,1-trichloroethane, experiencing a 9% increase in weight compared with 0.4% for the unexposed control. Another sealant, GE RTV60, increased in weight significantly when exposed to each of the three nonaqueous solutions:

- PF Degreaser 13%
- 1,1,1-Trichloroethane 5%
- Freon 113 28%

In contrast, specimens exposed to aqueous detergents averaged 0% change in weight with no large variation.

C.2.1.2 Analysis of LCA4 Full Exposure Matrix

There was a great deal of unexplained variability in the regression models from which these observations of main effects derived. This is due to variability associated with detergents and their interactions with adhesives, cleaning method, and temperature, as well as other factors that should be studied in subsequent efforts. To shed light on some of these effects, further modeling was performed. Analysis of the embedded design for Adhesive 3 (LCA4/BA5) and Detergent 1 (Versaclean) produced good models for change in weight following a single exposure and over the life cycle. These equations are shown below.

$$\Delta Wt_S = 0.494 - 0.119M_{UL} + 0.0642T - 0.0599f - 0.0442M_{UL}T - 0.0554\,M_{UH}T + 0.0064M_ST + 0.0248T^2$$

and

$$\Delta Wt_L = 0.157 - 0.218M_{UL} - 0.121f + 0.0600M_{UL}T + 0.070M_{UH}T + 0.180M_ST + 0.0603T^2$$

where M_i = indicator variable for cleaning method
T = temperature normalized from -1 to +1
f = sonication frequency; f = -1 for UL and +1 for UH

Each model tells us that percentage change in weight is a function of sonication frequency, the interaction of temperature and method, and the square of temperature. The average change in weight for this adhesive after a single exposure was 0.0270 percent compared to 0.0050 percent for the unexposed control. The extremes of the model account for variation in the range of -0.1 to 0.4 percent. The conclusion is that each is a very good model of dWt over the range exhibited; however, the total variation itself is not a significant phenomenon in the case of their adhesive-detergent combination. Further research is required to determine if these results generalize to other combinations.

C.2.2 Dimensional Changes

Table 13 in the main body provides summary statistics. Interesting specific results are presented below.

C.2.2.1 Sealants

Unexposed Ablestik 724/14C increased 8.4%. Larger increases were seen after exposure to Intex 8125, Oakite Liquid Detergent #2, PF Degreaser, 1,1,1-Trichloroethane, and Freon 113. Data on dimensional changes for PR1422B are sparse because of the degree of distortion. Values range from averages of -11.4% to 28.2% for various detergents. Similarly, GE RTV60 distorted and became spongy. Cleaning of these sealants by any means is not recommended.

C.2.2.2 Epoxies

The epoxies FA8/BA5, LCA9/BA5, and LCA4/BA5 showed no change. LCA4XM/BA5XM showed no change except for a 4.5% increase in combination with Intex 8125 and a 2.6% increase after exposure to Versaclean. For the remaining three epoxies, the following significant dimensional changes were observed:

- C-7/Activator W (0.5% for unexposed control)
  - Brulin 815 GD 2.4%
  - Alkaline-adjusted distilled water 2.7%

- Epon 828/Versamid 125 (control shrank 1.14%)
  - Brulin 815 GD 3.9%
  - MSI 1025 4.5%
  - PF Degreaser 6.5%
  - Citranox 9.5%

- Eccobond 2216 (2.1% increase for unexposed control)
  - MSI 1025 7.2%
  - Oakite Liquid Detergent #2 6.6%
  - Alkaline-adjusted distilled water 6.0%

Oakite Liquid Detergent #2 had a significant effect across adhesives and methods (4.03% increase compared to 0.2% for unexposed controls). Sonication at 67 kHz was associated with less of an increase in volume (0.6%) than immersion or 25 kHz sonication processes (1.6%). The highest temperature (195 F) was associated with a 2.3% increase in volume, which is statistically significant compared to the unexposed control.

C.2.3 Hardness

The adhesives evaluated in this study divided along predictable lines with respect to the hardness measure. The epoxies were harder than the sealants, with Shore D values in the 80 to 100 range for Adhesives 1-4 (FA8 and the LCAs). Adhesives 7, 9, and 10 (C-7, Epon 828, and Eccobond) were less hard but still had Shore D values in excess of 50. These adhesives maintained their hardness. Epon 828/Versamid 125 softened after initial exposure, but after life-cycle testing, was significantly harder than the unexposed controls. The statistics for each adhesive are shown in Table 13 (main body) for both single and life-cycle testing. Data for specific adhesive-detergent combinations may be found in Table C-3.

Table C-3. Shore Hardness Values for Adhesive-Detergent Combinations

	Adhesive										
Detergent	FA8/ BA5	LCA5/ BA5	LCA4/ BA5	LCA4XM BA5XM	Ablestik 124/14C	PR 1422B	C-7/ Act W	GE RTV60	Epon 828 Versamid 125	Eccobond 2216	Average by Detergent
Single Exposure											
Unexp. Control	80.1	89.0	88.0	87.6	82.7	16.1	73.4	32.6	79.0	64.2	74.5
Versaclean	89.2	97.8	86.6	85.9	87.5	NE	72.8	NE	80.6	56.0	84.9
Brulin 815GD	78.0	87.0	86.8	87.8	83.4	16.3	75.5	NE	71.4	62.7	75.4
EZE 240	87.7	93.7	91.7	85.6	88.5	NE	74.8	NE	76.0	59.4	81.7
Intex 8125	84.8	92.5	91.5	86.8	83.0	14.7	77.7	28.9	78.8	63.7	74.1
MSI 1025	76.7	92.0	88.3	87.6	86.7	NE	75.5	30.1	77.6	69.0	76.6
Oakite Liq Det #2	82.5	92.1	90.2	86.7	83.5	13.5	73.5	NE	77.3	64.6	78.4
PF Degreaser	NE	91.4	85.3	NE	81.7	22.7	78.8	21.5	78.1	NE	68.9
Citranox	87.0	92.6	87.2	83.4	88.0	NE	74.6	NE	75.8	66.7	81.9
Water	76.3	89.5	85.3	86.1	84.7	13.2	74.8	32.1	79.5	64.0	71.8
1,1,1-Trichloroethane	81.9	91.6	86.4	85.1	85.9	NE	71.9	29.0	78.9	61.8	75.9
Freon 113	77.8	88.5	88.9	87.4	84.8	23.0	77.4	29.0	77.1	65.3	72.4
Acidified Water	83.3	88.5	NE	84.2	86.3	NE	NE	NE	78.3	NE	84.0
Alkaline Water	NE	NE	89.4	NE	NE	NE	73.3	29.3	NE	61.2	65.0
Average by Adhesive	81.9	91.1	87.3	86.4	84.9	16.9	74.8	29.1	77.6	63.8	77.0

NE = Combination was not evaluated.

Table C-3. Shore Hardness Values for Adhesive-Detergent Combinations
(Continued)

Detergent	Adhesive										Average by Detergent
	FA8/ BA5	LCA5/ BA5	LCA4/ BA5	LCA4XM BA5XM	Ablestik 124/14C	PR 1422B	C-7/ Act W	GE RTV60	Epon 828 Versamid 125	Eccobond 2216	
Life Cycle											
Unexp. Control	80.7	89.8	86.4	84.4	85.9	29.3	71.3	35.4	73.1	64.7	73.7
Versaclean	84.1	95.8	86.0	85.9	88.3	NE	68.8	NE	75.7	47.1	83.4
Brulin 815GD	78.8	87.8	87.9	83.5	85.4	26.7	77.8	NE	67.8	61.4	75.7
EZE 240	86.9	93.4	88.4	84.7	87.3	NE	70.8	NE	77.5	68.5	82.1
Intex 8125	86.0	95.6	88.0	87.3	84.8	15.5	75.3	39.8	75.5	56.4	73.3
MSI 1025	79.3	92.9	86.1	84.7	87.3	NE	75.5	34.4	74.9	70.1	76.1
Oakite Liq Det #2	81.1	95.0	87.3	89.1	86.7	11.8	75.5	NE	77.0	64.4	77.9
PF Degreaser	NE	90.2	86.5	NE	85.7	22.8	79.1	19.2	79.3	NE	69.4
Citranox	79.2	97.4	87.8	84.4	87.9	NE	71.3	NE	73.8	72.4	81.7
Water	80.1	88.1	85.2	82.7	83.9	17.3	75.5	37.1	75.3	66.8	71.9
1,1,1-Trichloroethane	82.3	96.4	86.3	87.1	86.8	NE	71.4	36.1	69.6	54.0	74.4
Freon 113	80.4	90.1	87.0	85.5	86.6	15.0	73.4	37.2	78.6	61.9	72.4
Acidified Water	83.0	88.4	NE	84.3	88.1	NE	NE	NE	75.5	NE	83.8
Alkaline Water	NE	NE	86.9	NE	NE	NE	70.1	34.0	NE	70.5	65.3
Average by Adhesive	81.7	92.3	86.4	85.2	86.4	21.0	73.3	34.1	74.7	63.4	76.5

NE = Combination was not evaluated.

The sealants, Adhesives 6 (PR1422B) and 8 (GE RTV60), had lower values and their Shore A hardness tended to increase after 12 exposures. This was a function of aging, not of exposure, because the unexposed control specimens behaved similarly. PR 1422B survived a single exposure very well but softened and distorted considerably over the life-cycle.

C.3 Chemical Analysis

This section presents details of the analysis of change in glass transition temperature (T_g). It also provides some tutorial-type information about photoacoustic IR analysis and briefly describes the experimental procedure used in this study.

C.3.1 Analysis of T_g

C.3.1.1 Comparison of Life Cycle T_g with T_g for Single Exposure

The results for Adhesives 1, 2, 4, and 7 (FA8/BA5, LCA9/BA5, LCA4XM/BA5XM, and C-7/Activator W, respectively) indicate a significant increase in T_g over the life-cycle when compared with the result after a single exposure. For Adhesive 9 (Epon 828/Versamid 125), the change was not significant. For the remaining epoxy, Eccobond 2216, there was no basis for comparison with a single exposure but it did not differ significantly from the unexposed control.

When compared to the unexposed controls, Adhesive 1 (FA8/BA5) had a significantly higher T_g after a single exposure. Adhesive 2 (LCA9/BA5) showed no change after a single exposure, but a significantly higher T_g after the life cycle. This embrittlement indicates that it is adversely affected by some of the cleaning processes studied. Adhesive 3 (LCA4/BA5) showed a decrease in T_g over the life cycle. Adhesive 4 (LCA4XM/BA5XM) also exhibited a significant increase in T_g over the life cycle. The sealant tested, Adhesive 5

(Ablestik 724/14C), showed no change, nor did Adhesives 7 (Adhesive C-7/Activator W), 9 (Epon 828/Versamid 125), and 10 (Eccobond 2216).

Analysis of the effect of detergent on the difference in T_g over the life cycle indicated Intex 8125 was associated with a decrease (55.1 C to 47.6 C), while MSI 1025, Oakite Liquid Detergent #2, PF degreaser, Citranox, and distilled water were all associated with a significant increase in T_g. Table C-4 presents the statistical average T_g results after a single exposure and after the simulated life cycle. The right column, which shows the average by detergent, illustrates this point. On the other hand, there was no significant effect of the controls 1,1,-trichloroethane, Freon 113 or acidized distilled water. Similarly, there was no significant effect for Versaclean, Brulin 815 GD, and EZE 240. However, there was a great deal of variability in the results for EZE 240. This suggests the presence of interactions between factors, e.g., detergent and adhesive. These are explained in the next subsection.

For LCA9/BA5 and C-7/Activator W, there was a significant increase in T_g for the unexposed controls over the life cycle. This could indicate that these systems continue to cure over the life cycle.

C.3.1.2 Epoxy-Detergent Combinations

The paragraphs that follow discuss T_g effects for specific adhesives and relate them to particular detergent exposure. A detailed analysis was run on epoxy-detergent interactions. The results are shown in Table C-4. The numbers in the cells are the average values of T_g (C) for all samples of the indicated adhesive exposed (by any process) to the detergent listed at the left.

Table C-4. T_g Values for Epoxy-Detergent Combinations

Detergent	Epoxies: FA8/ BA5	LCA9/ BA5	LCA4/ BA5	LCA4XM BA5XM	C7/ Activator W	Epon 828/ Versamid 125	Eccobon d 2216	Average by Detergent
Single Exposure								
Unexp. Control	55.7	52.1	NE	NE	50.3	66.2	NE	56.1
Versaclean	51.0	50.4	NE	87.3	NE	63.4	NE	63.0
Brulin 815GD	NE	NE	NE	NE	48.9	NE	NE	48.9
EZE 240	61.5	60.9	NE	NE	NE	56.9	NE	59.8
Intex 8125	57.0	53.8	NE	54.5	NE	NE	NE	55.1
MSI 1025	NE	52.9	NE	NE	51.5	56.8	NE	53.7
Oakite Liq Det #2	55.5	54.7	NE	55.6	NE	57.9	NE	55.9
PF Degreaser	NE	NE	NE	NE	49.2	56.3	NE	52.7
Citranox	60.8	54.7	NE	NE	NE	58.4	NE	58.0
Water	NE	NE	NE	NE	50.1	NE	NE	50.1
1,1,1-Trichloroethane	53.4	54.1	NE	56.5	NE	NE	NE	54.7
Freon 113	NE	NE	NE	55.8	53.4	58.3	NE	55.8
Acidified Water	69.2	NE	NE	NE	NE	NE	NE	69.2
Alkaline Water	NE	NE	NE	NE	NE	NE	NE	NE
Average by Adhesive	58.0	54.2	NE	61.9	50.6	59.3	NE	56.7
Life Cycle								
Unexp. Control	54.9	62.6	96.3	63.3	64.3	63.8	18.9	59.5
Versaclean	95.4	85.9	84.0	NE	60.0	79.0	12.5	76.0
Brulin 815GD	NE	78.9	80.2	80.9	56.9	45.1	19.8	57.8
EZE 240	86.7	107.6	72.2	80.4	56.8	50.4	NE	73.9
Intex 8125	104.1	91.1	80.7	NE	25.0	50.3	19.3	47.6
MSI 1025	NE	88.6	79.6	81.4	63.7	60.2	NE	75.8
Oakite Liq Det #2	97.0	85.3	70.5	NE	65.1	57.5	NE	73.4
PF Degreaser	NE	77.3	78.3	NE	58.6	63.9	NE	69.5
Citranox	91.5	91.5	85.2	78.0	59.7	51.2	NE	74.3
Water	NE	94.0	85.1	84.6	62.3	51.0	NE	76.9
1,1,1-Trichloroethane	88.6	89.1	58.3	NE	61.2	63.6	20.9	63.3
Freon 113	NE	81.1	79.5	NE	70.1	53.2	20.0	54.0
Acidified Water	101.5	87.7	NE	83.0	NE	49.5	NE	80.4
Alkaline Water		NE	81.9	NE	49.8	NE	NE	60.5
Average by Adhesive	90.0	83.1	82.3	78.4	59.3	57.8	18.5	67.2

NE = Combination was not evaluated.

C.3.1.2.1 FA8/BA5. FA8/BA5 experiences a great increase in T_g (55.7 to 90.0 C) over the life cycle. The unexposed sample showed a Tg increase from 55 C to 112C. Therefore, any changes due to cleaning cannot be identified. This increase is least pronounced with 1,1,1-trichloroethane and EZE 240, but the difference is still significant. Therefore, cleaning of this epoxy by <u>any</u> of the means tested is not recommended if FA8/BA5 is used in an application where a significant increase in T_g is not acceptable. Typically, however, increased T_g is not a problem.

C.3.1.2.2 LCA9/BA5. In all cases, LCA9/BA5 exhibited a significantly higher T_g after exposure than the unexposed controls. Therefore, no counter indications were revealed unless the specific application cannot stand such a brittle material.

C.3.1.2.3 LCA4/BA5. LCA4/BA5, the most commonly used epoxy evaluated in this study, softened significantly upon exposure to Brulin 815 GD, EZE 240, Intex 8125, MSI 1025, Oakite Liquid Detergent #2 and PF degreaser, as well as 1,1,1-trichloroethane and Freon 113. Although it softened after exposure to the other solutions as well, the difference was not significant. Thus, these combinations are not counter indicated unless further testing reveals that such softening is unacceptable for the application under consideration.

C.3.1.2.4 LCA4XM/BA5XM. LCAXM/BA5XM showed a significant increase in T_g for all combinations evaluated.

C.3.1.2.5 C-7/Activator W. C-7/Activator W showed significant softening under many of the treatment conditions. Intex 8125 showed a particular poor result. T_g dropped to 24 C. Thus, this combination is not recommended. The following other detergents showed significant reduction in T_g for this epoxy: Versaclean, Brulin 815 GD, EZE 240, PF Degreaser, Citranox, and 1,1,1-trichloroethane. But these reductions were of the order of 2 to 7 degrees, which is probably not operationally important. The reasons such a small difference was statistically important was that the experimental error was so small for this adhesive. Use of these combinations is not recommended unless other factors warrant further evaluation in light of the application under consideration. Notably, water, MSI 1025,

and Oakite Liquid Detergent #2 caused no change, and Freon 113 was associated with a significant increase in T_g.

C.3.1.2.6 Epon 828/Versamid 125. Many cleaning solutions had a softening effect on Epon 828/Versamid 125. All but Versaclean, PF Degreaser, and 1,1,1-trichloroethane, including Freon 113, significantly decreased T_g. 1,1,1-trichloroethane and PF Degreaser had no effect while there was an increased T_g with Versaclean. Therefore, use with Versaclean is recommended for applications where an increase in T_g is acceptable, while PF degreaser and 1,1,1-trichloroethane appear to be acceptable.

C.3.1.2.7 Eccobond 2216. Eccobond 2216 is the softest of the epoxies. Its T_g value decreased significantly after exposure to Versaclean. Thus, this combination is not recommended. The combinations that showed no significant change are Brulin 815 GC, Intex 8124, 1,1,1-trichloroethane, and Freon 113.

C.3.1.3 Second-Order Model of LCA4/BA5

The analysis of the full factorial design on Adhesive 3 (LCA4/BA5) produced the following result:

$$\begin{aligned} T_g = \; & 82.5 - 0.15\, M_{UL} \cdot T + 11.1\, M_{UH} \cdot T \\ & + 13.3\, M_S \cdot T + 7.26\, T^2 \\ & - 1.06f - 4.00\, M_{UL}, \end{aligned}$$

where

M_i = an indicator variable for Method i
T = temperature normalized from -1 to +1
f = frequency; $f = -1$ for UL and $+1$ for UH.

The equation is a poor representation of the data, accounting for only 45 percent of the variability in the data. However, the most interesting result from this portion of the analysis is an apparent interaction between cleaning method and temperature. The second-order terms (interactions and T^2) are more influential than the main effects of these variables. More research is warranted to determine these effects.

C.3.2 Photoacoustic IR Analysis

C.3.2.1 Background

The photoacoustic affect was discovered by Alexander Graham Bell, who noticed that when a beam of sunlight shining onto an enclosed solid was periodically interrupted or modulated, a sound could be heard.[1,2] Others observed similar behavior when gases were exposed to modulated light.[3,4] While the photoacoustic effect with gases later became an accepted analysis technique, it was not until the 1970's that the photoacoustic effect with solids was accepted as a useful spectroscopic tool.

In its simplest form, photoacoustic spectroscopy (PAS) of solids involves placing the sample and a microphone in an enclosed cell with a window. Periodically chopped or modulated light is then allowed to impinge onto the sample producing sound. The sound, detected by the microphone, is at the same frequency as the chopping of the light. However, the amplitude of the sound is determined by the extent of absorption of the light by the solid. Therefore, if the frequency of the chopped light is varied and the amplitude of the audio signal is monitored an absorption spectrum of the sample can be obtained.

While PA spectra have been obtained in both the UV and visible regions, the mid-IR region is the most information rich in the electromagnetic spectrum for the study of coatings and adhesives. The mid-infrared region is where most organic materials have vibrational modes associated with their chemical bonds. The original work in PAS of solid substrates was conducted either with lasers or dispersive infrared spectrometers and had the problems of low throughput and low sensitivity associated with them. The advent of FT-IR

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[1] Bell, A. G.; Am. J. Sci., **1880**, 20, 305.

[2] Bell, A. G.; Philos. Mag., **1881**, 11, 510.

[3] Tyndall, J.; Proc. Roy. Soc. London, **1881**, 31, 307.

[4] Rontgen, W. C.; Philos. Mag., **1881**, 11, 308.

spectrometers coupled with photoacoustic detectors provided a tremendous improvement in sensitivity over these initial results. In this technique, a moving mirror apparatus, known as a Michelson interferometer, converts IR light into a superposition of IR frequencies, each modulated at a characteristic audio frequency. This modulated light is absorbed by the sample in the sealed sample chamber causing excitation of the vibrational modes of the chemical bonds in the sample. The decay of the excited state vibrational mode causes a thermal wave to pass to the sample surface where it creates a pressure wave which is detected by a sensitive microphone yielding the absorption spectrum of the solid coating or adhesive.

A tremendous advantage of the FT-IR over conventional dispersive IR spectroscopy is its ability to use all of the available radiant energy from the source at one time as opposed to one frequency at time in dispersive measurements. Since the FT-IR source is intense and already modulated, FT-IR is ideal for PAS experiments. The modulation frequency of the FT-IR, ω (rad/sec), varies as a function of IR frequency,

$$\omega = 4\pi V\nu$$

where V is the mirror velocity in centimeters and υ is the IR frequency in reciprocal centimeters.

The principal of photoacoustic effect is relatively simple. When a solid absorbs light, it becomes warm and heats the gas around it. In a closed vessel, the expansion caused by this effect leads to an increase in gas pressure. Turning off the light causes the temperature and pressure to return to the original values. Therefore, a modulated light source can cause a modulated pressure which can be detected as sound.

The photoacoustic signal depends strongly on the thermal diffusion length of the solid, μ_s. This quantity is a measure of the solid's ability to transfer heat to it's surface. Thus,

$$\mu_s = \sqrt{(2k/\rho C\omega)}$$

where k is the thermal conductivity of the solid, ρ is the density, C is the specific heat, and ω is the modulation frequency of the light (as discussed above). The effective sampling depth in PA FT-IR is μ_s because only those effects in the first diffusion length are important in determining the spectrum.

Quantitative analysis can be conducted with PA FT-IR although it is less straightforward than with transmission FT-IR spectroscopy. Typically a carbon black reference is used for ratioing the sample spectrum. This procedure removes instrumental artifacts, although an amplitude distortion is introduced by the variation in modulation frequency across the spectral range and the consequent variation in sampling depth. The use of an internal standard allows one to readily extract quantitative information. A calibration curve can also be created if the experiment is amenable to this type of treatment.

A number of PAS apparatus are available commercially. The Digilab division of BIORAD produces a PA cell as does EG&G PARC and MTEC Photoacoustics. Each provides a means to purge the sample with helium to improve signal-to-noise and to remove the IR absorbing water and CO_2.

C.3.2.2 Experimental Procedure

In a typical PA FT-IR spectroscopic experiment, each infrared spectrum consists of 400 co-added scans recorded at a 4 cm^{-1} resolution utilizing a mirror speed of 0.3 cm/sec corresponding to a modulation frequency of 5 kHz. All PA FT-IR spectra were recorded on a Digilab FTS-10M FT-IR spectrometer using a Digilab photoacoustic cell. The coadded sample scans were ratioed against a carbon black reference spectrum. All spectra were then transferred to an IBM-compatible personal computer for further spectral manipulation using Spectra Calc software (Galactic Industries).

Samples of the adhesives and detergents required little or no sample handling (simple placement into the sample cup was usually all that was required.) If the specimen was too large the excess was removed with a sharp knife or a straight-edged razor blade. In

the case of the lap shears, a sample was removed and used for analysis. Spectral subtractions were carried out to remove the effects of unchanged adhesive.

APPENDIX D

EXPERIMENTAL MATRIX AS RUN

Appendix D
Experimental Matrix as Run

The matrix design tables listed here describe the matrix trials as they were actually run. Please see Appendix A for an explanation of the variable coding used. Also, trials 108 and 88 could not be run at the specified temperatures. The specifications for these trials are listed below:

Trial	Adhesive	Detergent	Exposure	Temperature
108	4	7	S	3
88	1	7	UH	3

Trial	Group	Adhesive	Detergent	Cleaning Method	Temperature
1020	1	2	0	0	0
1070	1	7	0	0	0
1010	1	1	0	0	0
1	1	7	2	3	1
2	1	1	6	1	1
3	1	2	8	3	2
4	1	2	4	2	1
5	1	2	5	2	2
6	1	2	10	2	1
7	1	7	5	1	1
8	1	7	7	1	3
9	1	7	11	3	1
10	1	1	3	3	2
11	1	7	9	2	2
12	1	1	1	1	3
13	1	1	10	2	1
14	1	2	6	1	2
15	1	1	8	3	1
16	1	1	12	3	3
17	1	7	4	3	3
18	1	1	4	3	3
19	1	2	1	3	1
20	1	2	3	3	3

4 digit trial no.'s are unexposed controls; 1st is Group no.,
next two are adhes., with final no. of 0 being unexposed control

Trial	Group	Adhesive	Detergent	Cleaning Method	Temperature
2060	2	6	0	0	0
2090	2	9	0	0	0
21	2	10	8	2	1
22	2	10	7	3	3
23	2	10	3	1	2
24	2	10	5	3	1
25	2	10	9	3	2
26	2	10	6	3	1
27	2	10	13	2	3
28	2	9	7	3	2
29	2	9	3	1	1
30	2	6	2	2	3
31	2	9	6	2	3
32	2	6	4	3	2
33	2	9	5	1	3
34	2	9	11	2	1
35	2	9	8	3	3
36	2	9	1	3	2
37	2	6	7	1	2
38	2	6	9	2	1
39	2	6	6	3	3
40	2	6	11	3	1

4 digit trial no.'s are unexposed controls; 1st is Group no., next two are adhes., with final no. of 0 being unexposed control

Trial	Group	Adhesive	Detergent	Cleaning Method	Temperature
3030	3	3	0	0	0
3040	3	4	0	0	0
41	3	4	10	3	1
42	3	6	13	3	2
43	3	3	2	3	3
44	3	3	1	3	2
45	3	3	3	2	1
46	3	3	1	3	1
47	3	3	4	2	2
48	3	7	7	1	3
49	3	4	11	1	1
50	3	3	11	1	1
51	3	2	1	3	1
52	3	3	6	1	3
53	3	4	1	3	3
54	3	4	6	3	1
55	3	4	4	2	3
56	3	4	6	3	1
57	3	4	11	1	1
58	3	3	8	3	3
59	3	3	10	3	1

4 digit trial no.'s are unexposed controls; 1st is Group no.,
next two are adhes., with final no. of 0 being unexposed control

Trial	Group	Adhesive	Detergent	Cleaning Method	Temperature
4020	4	2	0	0	0
4050	4	5	0	0	0
4090	4	9	0	0	0
60	3	10	9	3	2
61	4	9	10	3	1
62	4	9	4	1	2
63	4	3	1	1	2
64	4	3	1	2	1
65	4	3	1	3	2
66	4	5	7	1	1
67	4	3	1	1	2
68	4	2	11	3	1
69	4	5	6	3	2
70	4	5	11	1	1
71	4	2	9	1	3
72	4	2	12	1	1
73	4	2	7	3	1
74	4	2	2	3	2
75	4	9	9	3	1
76	4	9	12	2	2
77	4	5	4	3	1
78	4	3	1	3	1
79	4	3	1	3	2
80	4	9	2	3	3
81	4	5	2	2	2

4 digit trial no.'s are unexposed controls; 1st is Group no.,
next two are adhes., with final no. of 0 being unexposed control

Trial	Group	Adhesive	Detergent	Cleaning Method	Temperature
5010	5	1	0	0	0
5030	5	3	0	0	0
5070	5	7	0	0	0
82	5	7	10	1	1
83	5	1	9	1	2
84	5	3	1	2	2
85	5	3	1	1	1
86	5	3	9	3	1
87	5	3	13	1	2
89	5	1	11	3	1
90	5	1	2	3	1
91	5	1	5	2	1
92	5	7	1	2	3
93	5	7	3	3	2
94	5	7	8	3	1
95	5	7	13	3	3
96	5	7	6	2	1
97	5	3	1	2	2
98	5	3	1	1	3
99	5	3	7	3	2
100	5	3	5	2	3
101	5	3	1	3	2
102	5	3	1	2	2

4 digit trial no.'s are unexposed controls; 1st is Group no.,
next two are adhes., with final no. of 0 being unexposed control

Trial	Group	Adhesive	Detergent	Cleaning Method	Temperature
6030	6	3	0	0	0
6040	6	4	0	0	0
6050	6	5	0	0	0
6100	6	10	0	0	0
103	6	10	4	1	3
104	6	10	1	3	3
105	6	10	10	3	1
106	6	10	2	1	1
107	6	10	11	2	1
109	6	4	5	3	1
110	6	5	8	1	2
111	6	5	9	3	3
112	6	5	12	3	1
113	6	4	12	1	3
114	6	4	8	1	1
115	6	4	9	3	2
116	6	4	3	2	2
117	6	4	2	2	1
118	6	3	1	1	2
119	6	3	1	2	3
120	6	3	1	3	3
121	6	5	3	2	3
122	6	5	1	2	1
123	6	5	5	3	2
124	6	5	10	3	1

4 digit trial no.'s are unexposed controls; 1st is Group no.,
next two are adhes., with final no. of 0 being unexposed control

APPENDIX E

LAP SHEAR EXPERIMENTAL DATA

Appendix E
Lap Shear Experimental Data

The following tables list the lap shear data arranged by trial order. Unexposed controls are listed by four digits. For example, 1020 is Group A (1), Adhesive 2 (02). The 0 at the end of the four digit number designates that trail as a control. Force to break is given in pounds. NA indicates the sample was not available to test. Lap shear trials 42, 46, 48, 49, 51, 56, and 60 all have zero measurements. Therefore, these trials are not listed in the following tables. Also, trials 108 and 88 could not be run at the specified temperatures. The specifications for these trials are listed below:

Trial	Adhesive	Detergent	Exposure	Temperature
108	4	7	S	3
88	1	7	UH	3

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables Group	Adhesive	Detergent	Cleaning Method	Temperature
1020	1926	2200	100	100	1	2	0	0	0
1020	2083	2240	100	100	1	2	0	0	0
1020	2457	2480	100	100	1	2	0	0	0
1070	3271	3689	90	90	1	7	0	0	0
1070	2358	NA	90	NA	1	7	0	0	0
1070	1940	3660	100	90	1	7	0	0	0
1010	3011	2633	60	60	1	1	0	0	0
1010	1697	2200	90	70	1	1	0	0	0
1010	2685	2260	50	90	1	1	0	0	0
1	3436	3067	70	100	1	7	2	3	1
1	3762	3067	70	90	1	7	2	3	1
1	4572	2800	90	60	1	7	2	3	1
2	1725	1877	100	60	1	1	6	1	1
2	1198	1892	100	100	1	1	6	1	1
2	1785	1692	100	100	1	1	6	1	1
3	2562	2100	100	100	1	2	8	3	2
3	2238	2450	90	100	1	2	8	3	2
3	2050	1600	100	100	1	2	8	3	2
4	NA	2255	NA	100	1	2	4	2	1
4	1940	2440	100	100	1	2	4	2	1
4	2313	NA	100	NA	1	2	4	2	1
5	NA	2073	NA	100	1	2	5	2	2
5	2313	1815	100	100	1	2	5	2	2
5	2420	2050	100	100	1	2	5	2	2
6	2064	1714	100	100	1	2	10	2	1
6	1815	1815	100	100	1	2	10	2	1
6	1886	NA	100	NA	1	2	10	2	1
7	3840	3455	60	90	1	7	5	1	1
7	3700	NA	60	NA	1	7	5	1	1
7	4000	3636	70	80	1	7	5	1	1
8	4075	NA	80	NA	1	7	7	1	3
8	3022	NA	90	NA	1	7	7	1	3
8	NA	NA	NA	NA	1	7	7	1	3
9	3617	2909	80	80	1	7	11	3	1
9	3111	2727	90	70	1	7	11	3	1
9	3571	2272	90	80	1	7	11	3	1
10	2721	2290	60	90	1	1	3	3	2
10	2177	1855	70	100	1	1	3	3	2
10	2286	1970	100	100	1	1	3	3	2

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables: Group	Adhesive	Detergent	Cleaning Method	Temperature
11	3800	NA	50	NA	1	7	9	2	2
11	2960	3083	70	60	1	7	9	2	2
11	3320	3362	60	90	1	7	9	2	2
12	1673	1917	100	80	1	1	1	1	3
12	2070	2473	90	100	1	1	1	1	3
12	2370	2433	80	80	1	1	1	1	3
13	2002	2364	95	70	1	1	10	2	1
13	2000	2436	80	80	1	1	10	2	1
13	2750	2200	80	80	1	1	10	2	1
14	2200	1631	100	100	1	2	6	1	2
14	2150	1800	100	100	1	2	6	1	2
14	2160	1952	100	100	1	2	6	1	2
15	2377	1667	85	100	1	1	8	3	1
15	2467	3455	90	80	1	1	8	3	1
15	2050	3500	95	70	1	1	8	3	1
16	1778	2327	95	80	1	1	12	3	3
16	2762	2185	100	80	1	1	12	3	3
16	1770	3060	95	70	1	1	12	3	3
17	2066	3050	90	50	1	7	4	3	3
17	1728	3100	95	70	1	7	4	3	3
17	1810	2357	90	NA	1	7	4	3	3
18	2222	3125	90	70	1	1	4	3	3
18	1944	2500	100	NA	1	1	4	3	3
18	2142	2167	90	95	1	1	4	3	3
19	2521	2167	100	100	1	2	1	3	1
19	2068	2146	100	100	1	2	1	3	1
19	2039	2300	95	100	1	2	1	3	1
20	2249	2000	60	90	1	2	3	3	3
20	2195	2109	50	100	1	2	3	3	3
20	2262	2750	100	80	1	2	3	3	3

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables: Group	Adhesive	Detergent	Cleaning Method	Temperature
2060	40	120	90	95	2	6	0	0	0
2060	140	164	90	95	2	6	0	0	0
2060	124	160	90	50	2	6	0	0	0
2090	4083	3320	50	50	2	9	0	0	0
2090	4636	4150	80	80	2	9	0	0	0
2090	4000	3167	80	80	2	9	0	0	0
21	3583	3382	50	50	2	10	8	2	1
21	3000	3520	60	50	2	10	8	2	1
21	NA	3333	NA	50	2	10	8	2	1
22	3200	NA	80	NA	2	10	7	3	3
22	3280	NA	50	NA	2	10	7	3	3
22	2920	NA	70	NA	2	10	7	3	3
23	2780	3217	80	90	2	10	3	1	2
23	3260	2817	50	50	2	10	3	1	2
23	2840	3164	90	50	2	10	3	1	2
24	3233	3417	50	95	2	10	5	3	1
24	NA	3077	NA	90	2	10	5	3	1
24	3060	3500	70	90	2	10	5	3	1
25	2741	2733	50	95	2	10	9	3	2
25	3167	2946	50	50	2	10	9	3	2
25	3036	1833	50	90	2	10	9	3	2
26	3273	3100	95	95	2	10	6	3	1
26	3436	3346	95	95	2	10	6	3	1
26	3164	3327	95	95	2	10	6	3	1
27	NA	3436	NA	90	2	10	13	2	3
27	3033	3563	60	80	2	10	13	2	3
27	3382	3050	90	90	2	10	13	2	3
28	3583	4000	NA	90	2	9	7	3	2
28	3800	3700	80	70	2	9	7	3	2
28	2867	4182	60	60	2	9	7	3	2
29	2655	3818	80	60	2	9	3	1	1
29	3420	3720	70	60	2	9	3	1	1
29	2960	3564	70	85	2	9	3	1	1
30	130	33	90	50	2	6	2	2	3
30	NA	40	NA	50	2	6	2	2	3
30	112	20	80	90	2	6	2	2	3
31	2933	3636	60	60	2	9	6	2	3
31	3458	2946	60	60	2	9	6	2	3
31	3610	2717	60	90	2	9	6	2	3
32	100	240	90	50	2	6	4	3	2
32	109	210	90	95	2	6	4	3	2
32	146	200	50	50	2	6	4	3	2

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables: Group	Adhesive	Detergent	Cleaning Method	Temperature
33	3430	2691	60	70	2	9	5	1	3
33	3561	3917	70	70	2	9	5	1	3
33	3200	4000	50	80	2	9	5	1	3
34	3700	4000	70	80	2	9	11	2	1
34	3250	2933	60	80	2	9	11	2	1
34	3955	3250	90	50	2	9	11	2	1
35	3800	3033	60	80	2	9	8	3	3
35	3035	3909	80	60	2	9	8	3	3
35	3782	3621	90	50	2	9	8	3	3
36	3361	3818	60	70	2	9	1	3	2
36	3417	3546	80	80	2	9	1	3	2
36	4467	4545	60	70	2	9	1	3	2
37	118	NA	80	NA	2	6	7	1	2
37	102	40	90	50	2	6	7	1	2
37	31	40	50	50	2	6	7	1	2
38	113	140	90	50	2	6	9	2	1
38	152	89.5	50	95	2	6	9	2	1
38	130	NA	90	NA	2	6	9	2	1
39	160	NA	50	NA	2	6	6	3	3
39	133	250	50	50	2	6	6	3	3
39	133	50	50	50	2	6	6	3	3
40	166	183	90	50	2	6	11	3	1
40	153	236	90	50	2	6	11	3	1
40	110	110	90	50	2	6	11	3	1

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables: Group	Adhesive	Detergent	Cleaning Method	Temperature
3030	2267	2300	70	80	3	3	0	0	0
3030	2250	2540	80	70	3	3	0	0	0
3030	2684	2382	70	60	3	3	0	0	0
3040	2688	2620	80	70	3	4	0	0	0
3040	2446	2660	70	60	3	4	0	0	0
3040	2527	2640	70	60	3	4	0	0	0
41	2300	2545	70	60	3	4	10	3	1
41	2364	2560	70	80	3	4	10	3	1
41	2300	2473	70	80	3	4	10	3	1
43	2273	2600	70	NA	3	3	2	3	3
43	2640	2860	60	NA	3	3	2	3	3
43	2473	2680	60	NA	3	3	2	3	3
44	2720	2800	70	50	3	3	1	3	2
44	2167	2640	80	50	3	3	1	3	2
44	2255	2780	60	70	3	3	1	3	2
45	2418	2800	60	70	3	3	3	2	1
45	2680	2673	80	60	3	3	3	2	1
45	2420	2564	70	80	3	3	3	2	1
47	2300	2267	60	80	3	3	4	2	2
47	2273	2383	60	70	3	3	4	2	2
47	2810	2250	60	50	3	3	4	2	2
50	2409	2400	70	70	3	3	11	1	1
50	2580	2473	60	70	3	3	11	1	1
50	2700	2546	80	70	3	3	11	1	1
52	2590	2760	70	70	3	3	6	1	3
52	2700	2800	70	60	3	3	6	1	3
52	2327	2582	90	80	3	3	6	1	3
53	2409	2436	60	80	3	4	1	3	3
53	2436	2640	80	80	3	4	1	3	3
53	2527	2655	70	70	3	4	1	3	3
54	2509	2473	70	60	3	4	6	3	1
54	2336	2680	60	80	3	4	6	3	1
54	2357	2436	70	80	3	4	6	3	1
55	2490	2540	60	70	3	4	4	2	3
55	2750	2769	70	80	3	4	4	2	3
55	2800	2436	60	70	3	4	4	2	3
57	2570	2680	70	80	3	4	11	1	1
57	2446	2509	60	70	3	4	11	1	1
57	2355	2473	70	70	3	4	11	1	1
58	2264	2660	70	70	3	3	8	3	3
58	2364	2560	60	80	3	3	8	3	3
58	2292	2267	70	80	3	3	8	3	3

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables Group	Adhesive	Detergent	Cleaning Method	Temperature
59	2409	2473	60	70	3	3	10	3	1
59	2455	2680	70	70	3	3	10	3	1
59	2446	2545	80	60	3	3	10	3	1

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables Group	Adhesive	Detergent	Cleaning Method	Temperature
4020	2360	2182	100	100	4	2	0	0	0
4020	2360	2236	100	100	4	2	0	0	0
4050	1333	862	95	100	4	5	0	0	0
4050	1400	1090	100	100	4	5	0	0	0
4090	3900	3833	50	80	4	9	0	0	0
4090	3600	4060	90	60	4	9	0	0	0
61	4156	3300	50	70	4	9	10	3	1
61	3840	3818	80	80	4	9	10	3	1
62	3346	2931	80	90	4	9	4	1	2
62	3218	2836	80	80	4	9	4	1	2
63	3022	2404	60	70	4	3	1	1	2
63	2800	2727	60	60	4	3	1	1	2
64	2708	2930	60	60	4	3	1	2	1
64	2833	2875	60	70	4	3	1	2	1
65	2833	2819	60	70	4	3	1	3	2
65	2800	2809	70	70	4	3	1	3	2
66	1360	1090	70	100	4	5	7	1	1
66	1360	1010	100	100	4	5	7	1	1
67	2958	2520	60	70	4	3	1	1	2
67	3300	2644	70	80	4	3	1	1	2
68	2300	3000	100	100	4	2	11	3	1
68	2280	1962	100	100	4	2	11	3	1
69	1280	782	100	100	4	5	6	3	2
69	880	920	100	100	4	5	6	3	2
70	1120	1140	100	100	4	5	11	1	1
70	1236	1160	100	100	4	5	11	1	1
71	2240	2250	100	70	4	2	9	1	3
71	1818	1873	100	100	4	2	9	1	3
72	2000	1979	100	100	4	2	12	1	1
72	2127	2458	100	100	4	2	12	1	1
73	2080	2140	100	100	4	2	7	3	1
73	2073	1690	100	100	4	2	7	3	1
74	1800	1920	100	100	4	2	2	3	2
74	1920	2375	100	60	4	2	2	3	2
75	3280	3560	80	50	4	9	9	3	1
75	3091	2940	70	80	4	9	9	3	1
76	3000	3273	90	50	4	9	12	2	2
76	3300	4100	70	70	4	9	12	2	2
77	1200	720	70	100	4	5	4	3	1
77	1360	900	100	100	4	5	4	3	1
78	2917	2340	60	70	4	3	1	3	1
78	2756	2380	80	80	4	3	1	3	1

Trial	Pounds to Break Single Exposure	Pounds to Break Life Cycle	% Adhesive Failure Single Exposure	% Adhesive Failure Life Cycle	Matrix Independent Variables Group	Adhesive	Detergent	Cleaning Method	Temperature
79	2792	2711	70	80	4	3	1	3	2
79	2792	2851	70	80	4	3	1	3	2
80	4100	2520	60	95	4	9	2	3	3
80	3822	2885	60	60	4	9	2	3	3
81	1160	780	85	100	4	5	2	2	2
81	1178	980	100	100	4	5	2	2	2

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables				
					Group	Adhesive	Detergent	Cleaning Method	Temperature
5010	2509	2618	95	90	5	1	0	0	0
5010	2160	2981	95	80	5	1	0	0	0
5030	2760	2854	70	80	5	3	0	0	0
5030	2720	2854	80	80	5	3	0	0	0
5070	3958	3689	50	60	5	7	0	0	0
5070	4300	3660	60	50	5	7	0	0	0
82	3604	3956	50	60	5	7	10	1	1
82	NA	4100	NA	60	5	7	10	1	1
83	3750	2773	90	70	5	1	9	1	2
83	2917	2208	90	100	5	1	9	1	2
84	2750	2917	70	70	5	3	1	2	2
84	2800	2720	70	70	5	3	1	2	2
85	2577	2854	80	80	5	3	1	1	1
85	2760	2640	70	70	5	3	1	1	1
86	2680	2720	80	80	5	3	9	3	1
86	2760	2800	70	70	5	3	9	3	1
87	2692	2640	70	80	5	3	13	1	2
87	2600	2760	70	80	5	3	13	1	2
89	2320	3500	92	70	5	1	11	3	1
89	2970	2020	80	100	5	1	11	3	1
90	3360	2417	70	80	5	1	2	3	1
90	3360	2417	60	100	5	1	2	3	1
91	3000	3167	95	95	5	1	5	2	1
91	3625	3440	60	70	5	1	5	2	1
92	4167	3447	70	50	5	7	1	2	3
92	3792	3440	60	50	5	7	1	2	3
93	3958	3727	60	70	5	7	3	3	2
93	3896	2792	60	70	5	7	3	3	2
94	3816	3787	70	60	5	7	8	3	1
94	4327	3542	70	60	5	7	8	3	1
95	3383	3061	60	50	5	7	13	3	3
95	3667	3280	60	60	5	7	13	3	3
96	4208	4000	70	60	5	7	6	2	1
96	4167	4156	80	60	5	7	6	2	1
97	2640	2458	70	70	5	3	1	2	2
97	2708	2660	70	80	5	3	1	2	2
98	2770	2280	70	70	5	3	1	1	3
98	2440	2680	80	70	5	3	1	1	3
99	2813	2540	60	80	5	3	7	3	2
99	2740	2760	80	70	5	3	7	3	2
100	2680	2345	80	70	5	3	5	2	3
100	2660	2540	80	90	5	3	5	2	3

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables Group	Adhesive	Detergent	Cleaning Method	Temperature
101	2460	2500	80	70	5	3	1	3	2
101	2520	2729	80	70	5	3	1	3	2
102	2680	2660	80	70	5	3	1	2	2
102	2750	2460	70	70	5	3	1	2	2

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables Group	Adhesive	Detergent	Cleaning Method	Temperature
6030	2660	2681	80	70	6	3	0	0	0
6030	2600	2667	50	80	6	3	0	0	0
6040	2560	2604	70	90	6	4	0	0	0
6040	2260	2510	70	70	6	4	0	0	0
6050	1229	1140	100	100	6	5	0	0	0
6050	1520	1100	60	100	6	5	0	0	0
6100	2542	3269	80	80	6	10	0	0	0
6100	2500	2292	70	50	6	10	0	0	0
103	2920	2000	60	50	6	10	4	1	3
103	2980	2800	80	80	6	10	4	1	3
104	3167	2875	50	60	6	10	1	3	3
104	3120	2960	50	50	6	10	1	3	3
105	2688	2833	60	50	6	10	10	3	1
105	2583	2320	60	50	6	10	10	3	1
106	2708	3280	50	60	6	10	2	1	1
106	3208	3160	50	50	6	10	2	1	1
107	3040	3444	50	50	6	10	11	2	1
107	2320	3260	60	50	6	10	11	2	1
109	2840	2660	80	70	6	4	5	3	1
109	2800	2600	80	70	6	4	5	3	1
110	1400	711	80	90	6	5	8	1	2
110	1000	820	100	100	6	5	8	1	2
111	708	440	100	100	6	5	9	3	3
111	708	327	100	100	6	5	9	3	3
112	1125	960	70	100	6	5	12	3	1
112	1240	800	100	100	6	5	12	3	1
113	3042	2708	60	80	6	4	12	1	3
113	2880	2760	80	80	6	4	12	1	3
114	2958	2750	80	70	6	4	8	1	1
114	2542	3067	80	80	6	4	8	1	1
115	2780	2500	60	60	6	4	9	3	2
115	2460	2770	80	80	6	4	9	3	2
116	2667	3156	70	70	6	4	3	2	2
116	2800	2541	70	80	6	4	3	2	2
117	2680	2720	70	80	6	4	2	2	1
117	2800	2571	70	70	6	4	2	2	1
118	2720	2700	60	80	6	3	1	1	2
118	2760	2872	80	70	6	3	1	1	2
119	2917	2820	70	80	6	3	1	2	3
119	2640	2917	70	70	6	3	1	2	3
120	2760	2960	70	70	6	3	1	3	3
120	2520	2865	60	80	6	3	1	3	3

Trial	Pounds to Single Exposure	Break Life Cycle	% Adhesive Single Exposure	Failure Life Cycle	Matrix Independent Variables Group	Adhesive	Detergent	Cleaning Method	Temperature
121	1313	896	60	100	6	5	3	2	3
121	1042	1000	100	100	6	5	3	2	3
122	1500	800	100	100	6	5	1	2	1
122	1174	1021	100	100	6	5	1	2	1
123	1080	625	100	100	6	5	5	3	2
123	917	851	100	100	6	5	5	3	2
124	969	489	100	100	6	5	10	3	1
124	1060	478	100	100	6	5	10	3	1

APPENDIX F

BULK SAMPLE EXPERIMENTAL DATA

Appendix F
Bulk Sample Experimental Data

The data listings that follow are the collected average values for bulk test results. They are arranged by adhesive type. The plaque number is an internal tracking code. The data and the units they are reported in are listed below.

Code	Description	Units
SSH	single exposure Shore hardness	Shore
LSH	life cycle exposure Shore hardness	Shore
SdV	single exposure % change in volume	%
LdV	life cycle exposure % change in volume	%
SdWt	single exposure weight change	%
LdWt	life cycle exposure weight change	%

				Responses					
Grp	Adh	Plaq	Trial No.	SSH	LSH	SdV	LdV	SdWt	LdWt
A	1	14	2	84.8	81.3	*	0.22	0.00	0.34
A	1	14	2	78.3	*	0.57	*	-0.01	*
A	1	16	2	84.5	80.8	*	0.40	-0.02	0.35
A	1	1	10	89.8	85.8	0.22	0.73	0.07	0.50
A	1	12	10	85.5	88.0	0.43	-1.29	0.07	0.54
A	1	8	12	*	*	0.08	*	0.09	*
A	1	8	12	91.3	85.0	*	0.04	0.02	0.67
A	1	9	12	87.0	83.2	*	0.38	0.01	0.62
A	1	4	13	84.5	82.0	*	1.84	0.01	0.36
A	1	10	13	75.0	*	-0.10	*	-0.01	*
A	1	10	13	86.3	82.5	*	-0.78	0.01	0.35
A	1	2	15	89.2	77.5	-0.78	-1.46	-0.02	0.33
A	1	3	15	84.8	80.8	0.00	0.26	-6.03	-5.68
A	1	6	16	87.3	81.7	1.42	0.54	0.32	0.98
A	1	15	16	76.0	*	-1.39	*	0.67	*
A	1	15	16	86.5	84.2	*	-0.05	0.31	0.98
A	1	5	18	87.0	84.2	-7.62	1.58	0.10	1.08
A	1	13	18	82.5	87.8	-0.56	-0.68	0.21	1.30
E	1	4	83	74.3	80.0	-0.03	1.08	0.01	0.32
E	1	8	83	78.3	80.2	-1.16	0.54	0.01	0.32
E	1	2	89	76.5	78.8	-3.89	-0.26	0.00	0.27
E	1	5	89	79.0	82.0	-3.44	-2.12	0.00	0.27
E	1	3	90	76.7	76.7	-1.28	-1.02	0.00	0.26
E	1	6	90	79.2	80.8	-1.74	-0.63	-0.03	0.24
E	1	9	91	77.2	79.8	-1.91	0.22	-0.01	0.28
E	1	12	91	76.2	78.7	1.36	1.64	-0.01	0.28
A	1	70	1010	75.7	*	*	*	-0.01	*
A	1	70	1010	83.5	81.0	*	0.18	0.00	0.18
A	1	110	1010	88.8	82.5	*	-0.94	0.00	0.19
E	1	100	5010	78.3	84.3	0.67	2.86	0.01	0.21
E	1	110	5010	74.3	74.8	-1.51	0.94	0.02	0.22

Grp	Adh	Plaq	Trial No.	Responses SSH	LSH	SdV	LdV	SdWt	LdWt
A	2	15	3	94.2	95.8	0.49	0.54	0.05	0.23
A	2	16	3	91.0	99.0	0.61	-1.58	0.05	0.24
A	2	11	4	96.7	99.0	*	-0.97	0.00	0.10
A	2	5	4	86.0	*	0.33	*	-0.02	*
A	2	5	4	94.7	92.2	*	-0.14	0.00	0.11
A	2	7	5	97.3	93.5	*	0.54	0.00	0.12
A	2	9	5	84.3	*	-0.56	*	-0.01	*
A	2	9	5	94.3	92.3	*	-0.87	0.00	0.10
A	2	14	6	94.7	96.2	*	0.11	0.00	0.10
A	2	8	6	86.0	*	-0.02	*	-0.01	*
A	2	8	6	94.0	96.5	*	0.78	0.00	0.11
A	2	10	14	95.7	94.5	*	-0.35	0.00	0.07
A	2	6	14	95.2	95.5	*	1.50	0.00	0.13
A	2	6	14	85.3	*	1.29	*	-0.01	*
A	2	1	19	100.0	93.8	-3.20	-0.84	0.00	0.10
A	2	2	19	95.5	96.7	0.18	-0.69	0.00	0.11
A	2	12	20	95.3	97.0	-2.96	-1.70	0.21	0.70
A	2	13	20	92.0	93.7	-2.80	1.50	0.21	0.66
D	2	1	68	88.3	90.7	-0.74	0.20	0.00	0.08
D	2	11	68	88.7	89.5	0.02	-0.68	0.00	0.08
D	2	5	71	90.7	89.7	0.24	0.34	0.05	0.22
D	2	7	71	88.3	86.5	0.14	0.04	0.21	0.39
D	2	3	72	89.7	89.8	-0.35	-0.46	0.00	0.05
D	2	8	72	87.3	87.0	-0.54	-0.28	-0.01	0.06
D	2	4	73	92.0	89.2	-0.46	-0.28	0.00	0.07
D	2	12	73	90.7	91.2	-0.29	0.50	0.00	0.07
D	2	6	74	85.5	87.8	-0.04	-0.25	0.04	0.20
D	2	9	74	88.5	87.8	-0.28	0.62	0.04	0.19
A	2	30	1020	79.7	*	-0.57	*	0.00	*
A	2	30	1020	94.7	92.3	*	-1.12	0.00	0.06
A	2	40	1020	94.2	90.3	*	0.36	0.00	0.07
D	2	100	4020	87.8	87.8	-1.92	0.06	0.00	0.06
D	2	200	4020	88.8	88.7	-0.58	-0.38	0.00	0.06

				Responses					
Grp	Adh	Plaq	Trial No.	SSH	LSH	SdV	LdV	SdWt	LdWt
C	3	11	43	89.7	88.5	0.18	1.20	0.12	0.40
C	3	12	43	83.8	87.2	0.03	-0.45	0.13	0.40
C	3	5	44	89.3	87.5	-0.04	0.08	0.04	0.19
C	3	9	44	87.3	87.0	-0.92	-0.64	0.04	0.19
C	3	14	45	92.8	88.8	0.07	-0.48	0.02	0.09
C	3	6	45	90.5	88.0	-0.22	0.20	0.01	0.08
C	3	10	47	93.2	89.2	0.48	-0.23	0.00	0.08
C	3	8	47	89.7	86.7	-0.09	-1.40	0.01	0.10
C	3	13	50	90.0	87.0	-0.21	-0.38	0.02	0.11
C	3	7	50	87.7	87.0	-0.13	-0.91	0.01	0.11
C	3	1	52	88.5	87.8	-0.26	-0.10	0.04	0.23
C	3	3	52	91.8	86.7	0.80	1.54	0.05	0.21
C	3	15	58	86.3	87.0	-0.13	0.45	0.16	0.24
C	3	16	58	88.0	88.5	-0.35	0.01	0.16	0.24
C	3	2	59	85.8	86.2	-0.75	-0.70	0.02	0.10
C	3	4	59	87.0	86.3	0.15	0.17	0.01	0.10
F	3	2	63	86.8	86.0	0.02	0.23	-0.07	-0.02
F	3	16	63	89.2	86.7	-0.02	-0.14	-0.00	0.08
F	3	1	64	84.7	88.2	0.03	0.55	-0.01	-0.05
F	3	10	64	86.0	83.7	-0.02	0.63	-0.01	0.05
F	3	4	65	85.0	90.2	0.49	0.84	0.02	0.13
F	3	18	65	84.7	84.5	0.00	0.16	0.02	0.12
F	3	9	67	84.3	84.2	0.57	0.46	-0.00	0.08
F	3	12	67	84.5	83.2	0.16	0.03	-0.01	0.07
F	3	7	78	82.3	88.8	-0.13	0.58	-0.00	0.06
F	3	11	78	85.7	88.5	-0.29	0.44	-0.01	0.06
F	3	3	79	84.3	86.3	-0.62	-0.30	0.36	0.13
F	3	13	79	85.0	83.8	0.48	1.22	0.02	0.13
E	3	12	84	85.3	86.7	-1.46	0.20	-0.01	0.05
E	3	21	84	85.5	87.0	-2.49	-0.46	-0.01	0.05
E	3	2	85	85.7	82.7	-0.52	0.55	-0.01	0.07
E	3	11	85	88.7	87.0	-1.02	0.35	-0.01	0.06
E	3	4	86	84.3	84.8	-0.89	0.26	-0.01	0.07
E	3	14	86	86.2	85.5	-0.44	1.01	-0.01	0.06
E	3	9	87	90.7	87.5	-1.43	0.41	0.00	0.08
E	3	15	87	88.0	86.2	-0.71	-0.01	-0.01	0.08
E	3	1	97	84.3	84.7	-0.67	1.35	-0.01	0.06
E	3	3	97	86.7	86.0	-1.43	0.46	-0.01	0.06
E	3	7	98	88.2	85.7	-1.04	0.31	0.04	0.18
E	3	17	98	85.8	86.2	18.20	21.17	0.04	0.19
E	3	10	99	84.7	86.2	-0.92	0.61	-0.01	0.05
E	3	20	99	85.8	86.7	-1.68	0.08	-0.01	0.06
E	3	13	100	87.2	86.5	-1.27	0.17	0.02	0.14
E	3	18	100	89.3	85.7	-2.00	0.15	0.02	0.13
E	3	5	101	85.3	87.7	-1.51	0.18	0.03	0.14
E	3	8	101	85.5	84.7	-1.23	1.21	0.02	0.14
E	3	16	102	89.3	88.8	-2.11	-0.08	-0.01	0.06
E	3	22	102	92.5	85.8	-0.54	0.96	-0.01	0.05
F	3	15	118	87.3	83.3	-0.26	0.13	-0.01	0.06
F	3	17	118	91.8	87.7	0.08	0.35	-0.00	0.07
F	3	5	119	90.5	86.2	-0.18	0.40	0.01	0.13
F	3	8	119	89.8	84.2	0.21	0.39	0.03	0.15

				Responses					
Grp	Adh	Plaq	Trial No.	SSH	LSH	SdV	LdV	SdWt	LdWt
F	3	6	120	85.0	85.0	0.26	0.47	0.13	0.42
F	3	14	123.5	84.3	82.8	0.26	1.24	0.15	0.42
C	3	170	3030	90.8	85.8	0.71	-0.06	*	0.09
C	3	180	3030	89.2	86.8	0.20	0.50	*	0.11
E	3	60	5030	86.0	87.2	-1.42	0.11	0.01	0.07
E	3	190	5030	85.8	85.8	-1.45	0.13	0.00	0.07

				Responses					
Grp	Adh	Plaq	Trial No.	SSH	LSH	SdV	LdV	SdWt	LdWt
C	4	1	41	85.3	85.5	-0.03	-0.03	0.01	-0.54
C	4	2	41	84.8	88.6	-0.03	0.00	0.01	0.12
C	4	10	53	86.5	84.5	0.13	0.85	0.22	0.57
C	4	9	53	85.2	87.3	3.58	4.43	0.19	0.52
C	4	5	54	88.7	89.2	0.40	0.65	0.01	-1.06
C	4	6	54	84.7	89.0	-0.91	0.73	0.00	0.11
C	4	4	55	86.7	87.8	-0.12	4.01	0.05	0.26
C	4	7	55	86.8	86.8	-1.89	5.02	0.07	-0.21
C	4	3	57	86.0	82.9	0.47	0.38	0.01	-0.37
C	4	8	57	88.8	88.0	0.00	0.20	0.02	0.11
F	4	12	109	87.5	85.5	1.58	0.89	-0.01	0.04
F	4	13	109	87.7	83.8	-0.20	0.41	-0.01	-2.66
F	4	6	113	83.5	84.8	0.28	0.66	-0.02	0.06
F	4	14	113	84.8	83.7	-0.09	0.43	-0.01	-0.01
F	4	1	114	83.0	83.8	0.23	0.46	-0.03	0.00
F	4	15	114	83.8	85.0	0.25	0.67	-0.04	-0.04
F	4	2	115	86.7	82.7	0.43	0.73	0.04	0.14
F	4	10	115	85.5	82.7	0.20	0.10	0.03	0.15
F	4	9	116	84.7	84.8	0.40	0.36	0.00	-0.17
F	4	16	116	86.5	84.5	4.19	0.67	0.04	0.08
F	4	4	117	89.3	84.2	-0.39	0.23	-0.02	0.04
F	4	8	117	86.3	82.7	-0.15	0.21	-0.01	0.04
C	4	110	3040	93.8	90.2	-0.10	4.04	0.00	0.09
C	4	120	3040	92.3	84.7	-0.34	0.49	0.00	0.10
F	4	3	6040	84.3	82.2	0.24	0.36	0.00	0.06
F	4	5	6040	83.3	83.3	-0.19	0.21	0.00	0.06
F	4	7	6040	86.7	82.5	-0.21	0.24	0.00	0.06
F	4	11	6040	85.0	83.5	-0.02	0.45	0.00	0.06

				Responses					
Grp	Adh	Plaq	Trial No.	SSH	LSH	SdV	LdV	SdWt	LdWt
D	5	3	66	82.7	87.7	2.80	9.05	0.52	0.55
D	5	9	66	80.7	83.7	1.27	10.48	0.45	0.50
D	5	4	69	81.3	85.5	1.39	8.88	0.67	1.32
D	5	8	69	85.7	87.8	3.19	10.21	0.65	1.31
D	5	11	70	85.2	86.7	3.64	8.32	0.50	0.49
D	5	12	70	84.3	86.5	3.76	11.91	0.52	0.47
D	5	7	77	83.2	84.3	4.24	11.46	0.47	1.29
D	5	10	77	82.8	85.2	2.46	10.14	0.55	1.35
D	5	2	81	83.0	84.7	3.72	10.83	0.53	1.00
D	5	6	81	83.7	86.0	-5.23	4.93	0.49	0.97
F	5	1	110	88.0	88.0	-0.30	0.79	-0.06	-0.04
F	5	6	110	88.0	87.8	-1.85	0.10	-0.03	-0.02
F	5	3	111	84.3	83.7	-1.09	0.49	0.36	0.28
F	5	8	111	85.0	84.0	-3.11	-2.84	0.39	0.24
F	5	4	112	85.0	88.2	-2.25	0.00	-0.02	0.01
F	5	10	112	87.5	88.0	-1.45	0.10	-0.02	0.06
F	5	7	121	88.5	87.3	-0.78	0.38	0.00	0.44
F	5	12	122	87.5	88.3	0.03	0.72	-0.02	0.04
F	5	5	123	86.3	86.3	-2.06	2.39	0.36	0.74
F	5	11	123	87.0	88.2	0.00	1.38	0.33	0.75
F	5	2	124	85.0	87.3	0.10	7.83	3.58	9.07
F	5	9	124	86.7	86.2	0.10	5.49	3.33	8.96
D	5	50	4050	84.2	87.5	2.01	9.00	0.49	0.45
D	5	100	4050	81.2	84.3	2.84	7.72	0.48	0.44

Grp	Adh	Plaq	Trial No.	Responses SSH	LSH	SdV	LdV	SdWt	LdWt
B	6	7	30	16.3	26.7	*	-8.35	-1.47	-4.26
B	6	1	32	14.7	15.5	*	-3.01	-0.86	-7.47
B	6	4	37	22.7	22.8	*	5.98	-0.83	-4.43
B	6	2	38	13.2	17.3	*	*	-1.00	-7.03
B	6	5	39	13.5	11.8	*	28.17	-5.00	-7.99
B	6	6	40	23.0	15.0	*	-0.30	-0.86	-8.14
B	6	30	2060	19.8	32.7	*	-16.49	*	-4.82
B	6	80	2060	12.3	25.8	*	-6.27	-2.71	-5.03

Grp	Adh	Plaq	Trial No.	Responses SSH	LSH	SdV	LdV	SdWt	LdWt
A	7	13	1	69.8	75.3	0.50	0.54	0.00	0.69
A	7	14	1	81.2	80.2	0.73	0.13	0.00	0.65
A	7	7	7	72.3	*	0.62	*	0.00	*
A	7	7	7	79.3	76.7	*	0.65	0.03	0.61
A	7	8	7	73.7	*	2.33	*	0.00	*
A	7	8	7	76.7	74.3	*	0.22	0.03	0.56
A	7	5	8	77.8	79.0	*	-0.36	0.04	0.75
A	7	6	8	79.7	79.2	*	-0.25	0.03	0.72
A	7	10	9	73.8	73.7	1.17	0.79	0.00	0.55
A	7	11	9	81.0	73.0	-0.10	-0.23	-0.01	0.66
A	7	12	11	72.3	*	-0.06	*	0.04	*
A	7	12	11	78.2	73.7	*	-0.08	0.03	0.69
A	7	9	11	72.3	*	0.05	0.38	0.02	*
A	7	9	11	76.3	77.2	*	0.72	0.03	0.65
A	7	2	17	75.2	73.6	0.76	2.95	0.74	4.24
A	7	3	17	80.2	77.0	1.38	1.92	0.75	4.30
E	7	1	82	72.2	72.5	0.03	0.38	0.25	0.62
E	7	6	82	71.5	70.3	0.07	0.28	0.22	0.60
E	7	4	92	71.8	68.7	0.52	2.07	0.43	1.69
E	7	10	92	73.7	68.8	0.51	1.64	0.39	1.59
E	7	7	93	73.3	69.8	0.02	0.97	0.39	1.23
E	7	12	93	76.2	71.7	-0.35	0.47	0.38	1.31
E	7	11	94	74.2	71.3	-0.52	0.22	0.27	0.62
E	7	13	94	75.0	71.2	-0.24	0.29	0.27	0.64
E	7	2	95	72.5	69.3	-0.14	2.33	1.18	3.75
E	7	14	95	74.0	70.8	0.41	3.04	1.18	3.78
E	7	3	96	72.2	74.0	-0.10	-0.70	0.22	0.59
E	7	5	96	74.7	77.0	0.30	0.52	0.21	0.55
A	7	40	1070	75.3	68.8	*	-0.38	0.03	0.52
A	7	40	1070	72.0	*	-1.16	*	0.04	*
A	7	100	1070	75.7	73.7	*	1.28	0.03	0.46
A	7	100	1070	70.3	*	0.30	*	0.03	*
E	7	80	5070	73.8	71.0	0.49	0.74	0.24	0.52
E	7	90	5070	73.3	71.8	0.23	0.34	0.24	0.52

				Responses					
Grp	Adh	Plaq	Trial No.	SSH	LSH	SdV	LdV	SdWt	LdWt
C	8	10	42	29.7	34.7	-0.98	-5.08	-0.37	-0.26
C	8	11	42	30.5	34.0	-1.75	-4.48	-0.38	-0.19
C	8	1	46	28.2	37.4	-1.79	-0.60	29.00	23.36
C	8	8	46	29.7	37.0	-1.22	-3.05	25.61	32.97
C	8	13	48	32.5	36.0	-0.59	-2.35	-0.32	0.85
C	8	9	48	31.7	38.2	0.60	-0.20	-0.25	-0.23
C	8	3	49	28.0	40.8	-0.20	-1.83	-0.22	0.68
C	8	7	49	29.8	38.8	-1.59	-4.17	-0.24	-0.43
C	8	12	51	30.0	34.0	-0.78	-4.12	-0.33	-0.05
C	8	5	51	28.5	34.0	-1.21	-2.82	-0.24	-0.16
C	8	4	56	29.5	37.5	0.41	-2.46	0.99	-1.44
C	8	6	56	28.5	34.7	-0.40	-1.61	0.95	11.51
C	8	14	60	22.2	17.0	0.60	-5.16	20.72	21.68
C	8	2	60	20.8	21.3	1.01	2.02	21.48	4.43
C	8	150	3080	34.0	36.2	*	-2.42	0.00	0.28
C	8	160	3080	31.2	34.5	*	-4.26	0.00	-0.28

Grp	Adh	Plaq	Trial No.	SSH	LSH	SdV	LdV	SdWt	LdWt
				Responses					
B	9	10	28	76.7	78.3	2.59	6.92	0.29	1.09
B	9	11	28	79.5	80.2	14.31	6.09	0.23	1.03
B	9	12	29	77.8	77.7	5.67	7.78	0.24	0.80
B	9	7	29	74.2	77.3	-7.20	-1.46	0.26	0.71
B	9	5	31	78.3	78.5	-5.57	3.12	0.97	1.95
B	9	9	31	76.3	75.5	0.55	0.57	0.47	1.62
B	9	15	33	76.3	76.0	0.94	10.16	0.47	1.56
B	9	6	33	78.8	73.8	-3.82	-1.12	0.43	0.90
B	9	14	34	75.8	77.2	0.96	2.25	0.23	0.73
B	9	4	34	78.3	80.0	0.23	-1.40	0.22	0.54
B	9	13	35	77.5	73.2	14.63	10.98	1.06	4.55
B	9	16	35	74.0	74.3	0.35	8.05	1.10	4.03
B	9	2	36	81.8	77.0	-6.27	-0.33	0.29	1.53
B	9	3	36	79.3	74.3	2.43	3.22	0.30	1.50
D	9	1	61	75.2	67.7	0.00	1.44	0.31	2.35
D	9	6	61	82.5	71.5	0.56	0.80	0.29	2.13
D	9	5	62	79.2	72.2	0.52	1.46	0.09	0.74
D	9	11	62	78.3	78.7	-0.69	0.14	0.10	0.91
D	9	3	75	78.8	73.7	0.34	0.65	0.04	0.54
D	9	7	75	80.2	76.8	-0.85	0.30	0.05	0.53
D	9	2	76	80.8	75.7	-0.39	-0.29	0.08	0.64
D	9	10	76	75.8	75.2	1.53	0.48	0.11	0.00
D	9	8	80	71.0	66.7	0.25	3.80	1.01	4.02
D	9	9	80	71.7	68.8	1.25	4.08	1.17	4.64
B	9	80	2090	82.2	76.5	*	-6.59	*	0.72
B	9	100	2090	81.3	76.0	*	3.46	0.00	0.35
D	9	40	4090	78.2	69.5	1.21	0.72	0.05	0.45
D	9	120	4090	74.3	70.5	-2.18	0.00	0.05	0.45

Grp	Adh	Plaq	Trial No.	Responses SSH	LSH	SdV	LdV	SdWt	LdWt
B	10	4	21	68.2	73.7	1.24	8.73	0.19	0.32
B	10	9	21	65.2	71.0	-3.83	0.29	0.17	0.31
B	10	1	23	51.3	66.8	2.64	7.36	0.18	0.58
B	10	7	23	67.5	70.2	-0.49	7.36	0.18	0.52
B	10	2	24	66.5	68.3	0.45	1.55	0.11	0.44
B	10	3	24	71.5	71.8	1.81	12.93	0.09	0.47
B	10	10	25	64.2	67.3	-3.87	2.44	0.24	0.73
B	10	11	25	63.7	66.2	1.98	-2.73	0.25	0.64
B	10	12	26	65.7	64.3	6.55	9.06	0.21	1.04
B	10	13	26	63.5	64.5	-3.20	4.13	0.32	1.03
B	10	14	27	68.5	71.7	*	10.48	*	0.46
B	10	5	27	67.8	69.2	*	1.56	-0.39	-0.04
F	10	6	103	63.2	55.2	-0.13	0.86	0.09	0.47
F	10	7	103	64.2	57.5	0.58	1.81	0.13	0.58
F	10	4	104	56.3	45.7	0.80	3.46	0.72	2.96
F	10	12	104	55.7	48.5	0.15	2.48	0.71	2.91
F	10	5	105	59.2	50.7	-0.22	0.85	0.64	2.78
F	10	9	105	64.3	57.3	0.68	2.10	0.64	2.78
F	10	3	106	62.2	61.0	1.13	0.77	0.11	0.19
F	10	11	106	63.2	61.7	0.13	0.28	0.01	0.15
F	10	1	107	67.8	61.2	0.84	1.70	-8.18	-8.04
F	10	10	107	62.7	62.5	0.90	0.53	0.00	0.17
B	10	60	2100	67.7	66.2	*	1.00	-0.34	-0.12
B	10	80	2100	67.3	68.2	*	9.24	*	0.38
B	10	150	2100	63.0	65.2	-1.93	3.43	0.55	1.34
B	10	160	2100	65.2	69.3	1.24	-0.90	0.44	0.67
F	10	80	6100	62.2	57.3	0.38	-0.13	0.01	0.16
F	10	200	6100	59.7	62.2	-0.01	0.11	0.01	0.18

Trial	Grp	Adh	Det	Meth	Temp	Plaque	Single	Life-Cy
1	1	7	2	3	1	13	48.9	56.9
2	1	1	6	1	1	14	55.5	97.0
3	1	2	8	3	2	15	54.7	91.5
4	1	2	4	2	1	5	53.8	91.1
5	1	2	5	2	2	7	52.9	88.6
6	1	2	10	2	1	8	54.1	89.1
7	1	7	5	1	1	7	51.5	63.7
8	1	7	7	1	3	5	49.2	58.6
9	1	7	11	3	1	10	53.4	70.1
10	1	1	3	3	2	1	61.5	86.7
11	1	7	9	2	2	9	50.1	62.3
12	1	1	1	1	3	8	51.0	95.4
13	1	1	10	2	1	4	53.4	88.6
14	1	2	6	1	2	6	54.7	85.3
15	1	1	8	3	1	2	60.8	91.5
16	1	1	12	3	3	6	69.2	101.5
17	1	7	4	3	3	3		25.0
18	1	1	4	3	3	5	57.0	104.1
19	1	2	1	3	1	1	50.4	85.9
20	1	2	3	3	3	12	60.9	107.6
1010	1	1	0	0	0	70	55.7	54.9
1020	1	2	0	0	0	30	52.1	61.3
1070	1	7	0	0	0	10	50.3	61.4

Trial	Grp	Adh	Det	Meth	Temp	Plaque	Single	Life-Cy
28	2	9	7	3	2	10	56.3	63.9
29	2	9	3	1	1	7	56.9	50.4
31	2	9	6	2	3	5	57.9	57.5
33	2	9	5	1	3	15	56.8	60.2
34	2	9	11	2	1	4	58.3	53.2
35	2	9	8	3	3	13	58.4	51.2
36	2	9	1	3	2	3	63.4	79.0
2090	2	9	0	0	0	100	66.2	61.6

Trial	Grp	Adh	Det	Meth	Temp	Plaque	Single	Life-Cy
41	3	4	10	3	1	1	56.5	
43	3	3	2	3	1	11		80.2
44	3	3	1	3	2	5		58.3
45	3	3	3	2	1	6		72.2
47	3	3	4	2	2	10		80.7
50	3	3	11	1	1	13		79.5
52	3	3	6	1	3	1		70.5
53	3	4	1	3	3	10	87.3	
54	3	4	6	3	1	5	55.6	
55	3	4	4	2	3	7	54.5	
57	3	4	11	1	1	3	55.8	
58	3	3	8	3	3	13		85.2
59	3	3	10	3	1	4		58.3
61	4	9	10	3	1	1		63.6
62	4	9	4	1	2	5		50.3
63	4	3	1	1	2	2		90.5
64	4	3	1	2	1	1		78.8
65	4	3	1	3	2	4		82.7
65	4	3	1	3	2	4		94.5
67	4	3	1	1	2	9		77.6
68	4	2	11	3	1	1		81.1
69	4	5	6	3	2	8		-16.4
70	4	5	11	1	1	12		-30.0
71	4	2	9	1	3	5		94.0
72	4	2	12	1	1	3		87.7
73	4	2	7	3	1	4		77.3
74	4	2	2	3	2	6		78.9
75	4	9	9	3	1	3		51.0
76	4	9	12	2	2	2		49.5
77	4	5	4	3	1	10		-27.5
78	4	3	1	3	1	0		81.0
79	4	3	1	3	2	3		80.2
80	4	9	2	3	3	8		45.1
81	4	5	2	2	2	6		-26.9
4020	4	2	0	0	0	200		71.8
4020	4	2	0	0	0	200		54.6
4050	4	5	0	0	0	100		-26.2
4090	4	9	0	0	0	120		65.2
4090	4	9	0	0	0	120		64.7

Trial	Grp	Adh	Det	Meth	Temp	Plaque	Single	Life-Cy
82	5	7	10	1	1	1		62.0
82	5	7	10	1	1	6		60.4
84	5	3	1	2	2	12		81.5
85	5	3	1	1	1	11		81.1
86	5	3	9	3	1	4		85.1
87	5	3	13	1	2	15		81.9
92	5	7	1	2	3	4		61.2
92	5	7	1	2	3	10		58.7
93	5	7	3	3	2	7		55.9
93	5	7	3	3	2	12		57.6
94	5	7	8	3	1	11		56.8
94	5	7	8	3	1	13		62.5
95	5	7	13	3	3	2		51.0
95	5	7	13	3	3	14		48.5
96	5	7	6	2	1	3		67.9
96	5	7	6	2	1	5		62.3
98	5	3	1	1	3	7		80.8
99	5	3	7	3	2	10		78.3
100	5	3	5	2	3	13		79.6
101	5	3	1	3	2	5		87.5
102	5	3	1	2	2	16		78.8
5030	5	3	0	0	0	60		103.3
5070	5	7	0	0	0	80		67.1
5070	5	7	0	0	0	80		65.5
5070	5	7	0	0	0	90		61.4
5070	5	7	0	0	0	90		66.0

Trial	Grp	Adh	Det	Meth	Temp	Plaque	Single	Life-Cy
103	6	10	4	1	3	6		22.7
103	6	10	4	1	3	6		18.7
103	6	10	4	1	3	7		15.0
103	6	10	4	1	3	7		20.6
104	6	10	1	3	3	4		14.2
104	6	10	1	3	3	12		10.8
105	6	10	10	3	1	9		20.9
106	6	10	2	1	1	3		18.3
106	6	10	2	1	1	11		21.3
107	6	10	11	2	1	1		21.3
107	6	10	11	2	1	10		18.7
109	6	4	5	3	1	12		82.7
109	6	4	5	3	1	13		80.0
110	6	5	8	1	2	1		-29.8
111	6	5	9	3	3	3		-11.8
112	6	5	12	3	1	4		-30.7
112	6	5	12	3	1	10		-27.5
113	6	4	12	1	3	14		83.0
114	6	4	8	1	1	1		75.7
114	6	4	8	1	1	15		80.2
115	6	4	9	3	2	2		91.4
115	6	4	9	3	2	10		77.8
116	6	4	3	2	2	9		80.5
116	6	4	3	2	2	16		80.2
117	6	4	2	2	1	4		82.7
117	6	4	2	2	1	8		79.0
118	6	3	1	1	2	17		81.9
119	6	3	1	2	3	8		101.0
120	6	3	1	3	3	14		107.5
121	6	5	3	2	3	7		-24.1
122	6	5	1	2	1	12		-30.0
123	6	5	5	3	2	11		-26.5
124	6	5	10	3	1	2		-19.2
124	6	5	10	3	1	9		-27.2
6030	6	3	0	0	0	170		89.2
6040	6	4	0	0	0	70		64.3
6040	6	4	0	0	0	110		62.2
6100	6	10	0	0	0	80		24.7
6100	6	10	0	0	0	200		14.6
6100	6	10	0	0	0	200		17.5

APPENDIX G

COMPLETE LIST OF DETERGENT CANDIDATES

Appendix G
Complete List of Detergent Candidates

The tables in this appendix list assorted technical specifications for the various detergents, as well as their respective manufacturers.

Table G-1. Surface Tension of Detergent Solutions

Detergent	Detergent	Surface Tension, dynes/cm^2
1	Versaclean	34.0
2	Brulin 815GD	33.3
3	EZE 240	35.4
4	Intex 8125	34.7
5	MSI 1025	35.1
6	Oakite Liq. Det. #2	30.0
7	PF Degreaser	27.3
8	Citranox	34.7
C_1	Distilled Water	73.5
C_2	1,1,1-Trichloroethane	31.0
C_3	Freon 113	22.9
C_4	Acid	57.0
C_4	Base	59.6

Standard concentration at 25 C.

Table G-2. pH Stability of Detergent Solutions

Detergent Number	Initial pH(A)	Post Life Cycle pH(B)
1	9.62	10.14
2	10.32	10.39
3	8.98	8.87
4	8.51	8.96
5	8.50	9.44
6	8.94	9.57
7(C)	--	Det. # 7 not run at 190 F
8	2.94	3.37
C_1	4.27	6.73
C_4A	2.00	1.90
C_4B	12.06	12.83

(A) Standard concentrations, diluted for use

(B) Life cycle immersion at 190 F

(C) Detergent 7 is PF degreaser, a mixed hydrocarbon. It does not have a pH which is comparable to the other water based detergents.

Product Name	Supplier	AGMC Production Usage	Product Class/Family	Matrix Status	Alkaline Fortified	Phosphate Content	Solvent Status/Type of Solvent	Comment
				Brulin Company				
815GD	Brulin	No			Yes	Yes		Product is used in other aerospace applications, GD stands for General Dynamic
815QR	Brulin	No	Formulated detergent containing phosphates and amines	Maybe	Yes	Yes	Unknown, no flashpoint, no MSDS	Described as an extra strength general purpose cleaner and degreaser
815QD	do not have information on this product							
				DuBois				
L-4000	DuBois	No	Alkaline cleaner	Recommend	Yes	Yes	Unknown, no flashpoint, no MSDS	Listed as an electronic circuit cleaner, recommended for flux removal and circuit cleaning; passes MIL-P-28809
				EZE Products, Inc.				
EZE 246A	EZE Products, Inc.	No	Formulated mixed surfactants		No	Unknown	Yes, diethylene glycol monobutylether	Supposed to out perform the alkaline cleaners in ultrasonic batch
EZE 240	same	No	Formulated surfactant product	Not recommended; will leave residues	No	Unknown	Yes, hexylene glycol and ethanolamine	Designed for cleaning metals which easily flash rust; contains rust and antioxidants as additives
EZE 425	same	No	Formulated mixed surfactants	Not recommended	No	No; unknown	Yes, hexylene glycol, 147 F flash	Cleaning booster; for use with alkaline
Intex 8215	Intex Chemical, Div of EZE Products	No	Solvent based rust preventative	Not recommended	No	No	Yes, hydrocarbons at 55-60; dipropylene glycolmethyl ether 1-3; 187 F flash	Product is a rust preventative, not tested on aluminum, very high solvent content
Intex 8125	Intex Chemical, Div of EZE Products	No	Solvent	AGMC recommended	Yes, pH neutral by neutralizing	Unknown	Yes, dipropylene glycol methylether	Designed for ultrasonic and soak tank use, especially designed to prevent dissimilar metal reactions

Product Name	Supplier	AGMC Production Usage	Product Class/Family	Matrix Status	Alkaline Fortified	Phosphate Content	Solvent Status/Type of Solvent	Comment
					Magnasonic			
MSI 1025	Magnasonic; BCD Inc.	Yes		Recommended	Unknown	Unknown	Unknown	Instrument bearing cleaning
	Magnasonic; BCD Inc.	Yes	Not being used as a cleaning solution	Recommended; AGMC no	Unknown	Unknown	Unknown	Flux remover, studied previously by Phil Schmacher
MSI 8700	Magnasonic; BCD Inc.	Yes		Recommended	Yes, from G. Spriggs liquid version of MSI 1067	Unknown	Unknown	Scale remover for tubes and prism cleaning; 9.5 pH with buffering additives
					Union Carbide			
Tergitol 15-S-9	Union Carbide	No	Nonionic, ethylene oxide adduct with secondary alcohols	Recommended as nonproduction nonionic system	No	Zero	None	Non-formulated straight nonionic which is recommended for electronic circuit board cleaning
Tergitol 15-S-7	Union Carbide	No	Same, only 7 EO units	Not recommended; product line already represented	No	Zero	None	Non-formulated straight nonionic which is recommended for electronic circuit board cleaning
					Simple Green			
Simple Green	Simple Green	No	Nonionic		No	No	Yes, butyl cellosolve	Established product presence of butyl cellosolve is questionable
					P-T Technologies			
PF Degreaser	P-T Technologies	Yes	Nonhalogenated, low boiling hydrocarbon mixture	Recommended				Review matrix status-product is really a solvent that is sold as a "safe" replacement for 1,1,1 and freon; highly volatile hydrocarbon

Product Name	Supplier	AGMC Production Usage	Product Class/Family	Matrix Status	Alkaline Fortified	Phosphate Content	Solvent Status/Type of Solvent	Comment
					Oakite			
Liquid Detergent #2	Oakite	Yes		Recommended	Yes, mold rating by manufacturing, pH 10.5 at standard use level	None	Unknown, some high boiling diluent is presence which may act as a solvent, but no flash point created	Surfactants chelates alkaline salts, need MSDS sheet
					Mirachem Corporation			
100 Cleaner/Degreaser	Mirachem Corp.	No		Not recommended	cannot find product data			
250 Rust and scale remover	Mirachem Corp.	No	Maybe	Not recommended	No	cannot find product data		Acidic scale and rust remover
					Hurri Klean			
Hurri Safe Special Formula	Hurri Klean	No	Alkaline degreaser		Yes	Unknown, not listed in company literature	Yes, butyl cellosolve ~ 10 percent	Recommended for electronic cleaning
					Rose Chemical			
Rose Tergo	Rose Chemical	No	Anionic formulated with amides		No	Yes	No	Supplier gives very complete formulation details in MSDS
					W. R. Grace			
Dara Clean 220	W. R. Grace	No	no other information					
282		No	Alkaline all-purpose cleaner	Recommended	Yes	No	Yes, unspecified glycol ethers	Recommended for electronic cleaning and Al and Zn compatibility
283		No	Alkaline all-purpose cleaner		Yes	No	Unknown	Multimetal safe, limited to 150 F use, this suggests a solvent component low foam for spray application